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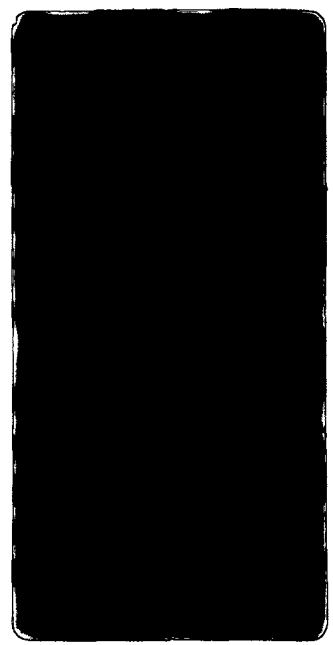

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FINAL REPORT

"Fabrication and Properties of Tungsten and Tungsten Alloy Single Crystals"

by the Staff of

Linde Company

and

Haynes Stellite Company

Divisions of Union Carbide Corporation

For period June 29, 1961 - February 28, 1962

Contract: NOw 61-0671-C

For the Materials Branch,

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PREFACE

This work was done by the staffs of the Linde Company and Haynes Stellite Company with Milton Stern as principle investigator. At the Linde Company, R. W. Diesner conducted the majority of the experiments including metallurgical observations, special heat-treatments, and recrystallization measurements; L. G. Tensmeyer and R. G. Rudness prepared the various special composition crystals. At the Haynes Stellite Company, J. W. Chasteen supervised fabrication and sample preparation for mechanical property measurements.

Early work described in the introduction was obtained in a cooperative program between the Linde Company and Union Carbide Metals Company. These data were obtained by E. L. Harmon, J. L. Wilson and R. W. Fountain.

SUMMARY

This is the final report of Contract NOw 61-0671-C and covers the period June 29, 1961 to February 28, 1962. The objective was to evaluate the fabricability and properties of tungsten and tungsten alloy single crystals. This was accomplished with sixty-five crystals prepared with diameters ranging from 1/2 to 1 inch and lengths from 6 to 18 inches. Included in the program are several dilute alloys. The majority of crystals have been successfully swaged, forged or rolled at temperatures far below those required to fabricate polycrystalline tungsten. The following properties were determined on the fabricated material: hot hardness, recrystallization temperature, mechanical properties at elevated temperatures, and ductile to brittle transition temperature.

It is shown that single crystals have a significant fabrication advantage and that their mechanical properties after working are equivalent to material of similar composition prepared by other commercial processes. Surface preparation is important in obtaining good fabrication and low bend ductile-brittle transition temperatures. The single crystals were extensively characterized by chemical analyses and X-ray and metallographic observations.

I. INTRODUCTION

A. Background

Many present and future materials problems require performance under extreme conditions of temperature and environment. This has led to extensive work involving tungsten and tungsten base alloys since the high melting point of these materials promises attractive strength properties if other problems specific to the material can be effectively solved. It is worth noting that some of the properties of tungsten which make it attractive for future application are often the same properties which create difficulty in furthering the application of this material. Major problems today involve efficient consolidation into dense metallic form, fabrication into useful shapes, and creation of unusually strong materials while minimizing the ductile to brittle transition temperature.

Consolidation of tungsten is primarily achieved by four different techniques: pressing and sintering, slip casting, consumable arc melting, and arc torch spraying. Electron beam and induction heated floating zone refined crystals have also been produced; however, the maximum size attainable by this approach is quite small and hence their application will probably be quite limited. While each process produces final materials and shapes with some utility, many factors must be significantly improved before a completely satisfactory general process is achieved.

This work describes tungsten materials prepared in single crystal form by an arc modification of the Verneuil process.

B. Fabrication

Today's techniques provide either low density, poor composition control, very poor mechanical properties, or low ductility. Also, they uniformly produce material which is very difficult to fabricate without extremely high temperatures and high deformation rates.[1] Consumable arc-melted tungsten requires extrusion at high deformation rates to break up the large grain size of the cast structure.[2] Even after such treatment, swaging must be done at temperatures as high as 1450°C (2640°F). Direct swaging of arc-melted tungsten requires temperatures in excess of 1700°C (3090°F).[3] Tungsten produced by powder metallurgical methods is swaged at temperatures of about 1500°C (2730°F). Hammer forging is started at about 1800°C (3270°F) with sheet rolling becoming practical at 1300°C (2370°F) to 1400°C (2550°F) after some direct forging for initial breakdown.[1]

The extreme conditions required to process tungsten are multiplied many fold for tungsten base alloys with much greater

strengths at high temperatures. The technical possibility of creating tungsten alloys with significantly improved strength properties over tungsten has been established.[4] However, the utility of such materials will never become a reality unless processing conditions which are realistic and reasonable can be established for their fabrication.

This program describes an approach with a high probability of success for achieving conditions which will permit ready fabrication of very high strength tungsten base alloys. The approach utilizes the improved fabricability of single crystals. The ease of deformation of single crystals has been realized for a number of years.[5] It has only recently been possible to produce massive single crystals of tungsten and its alloys.[6]

Samples of very small diameter tungsten single crystals grown by the strain anneal method have been known for some time[7] although property information has been limited and subject to some doubt because of incomplete characterization of the material. While extensive physical and mechanical property data for polycrystalline tungsten are reported[8] recent data on large single crystals have not been available. Some preliminary data obtained on a cooperative program within the Union Carbide Corporation between the Linde Company and the Union Carbide Metals Company show some rather interesting and surprising effects on fabricability of tungsten single crystals. Table I presents results of swaging tests on as-grown crystals of various orientation. The tests were done on material of approximately 1/2-inch diameter. The orientations* indicated represent the crystallographic direction along the axis of the cylindrical sample.

Swaging at 1000°C (1830°F) presented no problem. At 700°C (1290°F) partial reduction occurred easily but rapid cooling in the massive swaging machine caused a sufficient temperature drop to result in fracture of the samples. Since it is possible that as-grown crystals contain stresses which could influence their fabricability,[9] a few swaging tests were conducted after a stress relief at 1700°C (3090°F).

The tests recorded in Table II illustrate a remarkable difference in the fabricability of single crystal and polycrystalline tungsten. Single crystal material can be worked at from 500° to 700°C (900° to 1360°F) lower than conventional material. This is perhaps not surprising when one considers all the evidence for impurity segregation and second phase precipitation at grain boundaries.

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* The numbers in brackets [ ] are crystallographic directions; the numbers in parenthesis ( ) are crystallographic planes. Since tungsten is body centered cubic, the direction [a b c] is perpendicular to the plane (a b c).

C. Mechanical Property Relations

Considerable data are now available on the mechanical properties of tungsten produced by various techniques.[10-14] Table III summarizes some of the pertinent data. The comparison is not yet complete or exact, since it is quite certain that impurities and surface preparation play a prominent role in determining mechanical properties - particularly at the lower temperatures. It is clear, however, that high purity single crystals have ductile to brittle transition temperatures well below room temperature when the surface is properly prepared. Also single crystals have high temperature strength properties comparable to polycrystalline tungsten of similar purity.

Table IV shows mechanical property data for a few polycrystalline tungsten alloys tested at 1650°C ($\approx$ 3000°F). These alloys are among the strongest reported today at very high temperatures. Only a few metallic materials are in the same strength region as the W-0.6% Nb alloy; a W-30% Re alloy,[15] single crystal tungsten containing 2% ThO_2 as a dispersed phase,[8] and a 88.6% W-5.7% Mo-5.7% Nb alloy.[16]

D. Single Crystal Program

From Tables I to IV, it is evident that the strength and ductility of Linde single crystal tungsten is about equal to or better than tungsten prepared by various techniques. One problem in the comparison of properties is the establishment of sound base line data. Two important variables are surface conditioning and chemical composition. Stephens found a pronounced increase in ductility by removal of the outer surface of tungsten rods.[17] Surface removal was found to have a pronounced effect upon the fabricability and ductile-brittle bend transition temperature of the tungsten crystals studied.

The effect of interstitial elements and metallic impurities is also important[10][18][19]. One of the more extensive studies made was the effect of chemical composition. The individual effects of the alloying elements was studied and chemical analyses of all successfully worked material were obtained. In this way it was felt that the material would be well characterized. Generally, it was found that the starting crystal had lost some of the alloying agent during growth and that the carbon and the oxygen were both less than 20 ppm (in some cases less than 10 ppm).

With the material well characterized regarding carbon, oxygen, metallic impurities and alloying agents, consistent experimental procedures were carried out. In this way it was hoped to isolate the effects of alloying elements, crystallographic orientation, and crystal perfection. These were studied on the basis of fabricability, tensile strength, hot hardness, recrystallization temperatures, room tem-

perature hardness and some bend ductility tests. All of the specimens were also examined metallographically.

EXPERIMENTAL PROCEDURES

A. Crystal Growth

Tungsten and tungsten alloy crystals were grown by an arc modification of the Verneuil technique. The basic Verneuil method is described by Buckley.[20] Sixty-five crystals were prepared for this program. Their diameters ranged from 1/2 to 1-1/8 inch with lengths varying from 6-18 inches. Most unalloyed crystals were 12 inches long. However, the alloy crystals and the high purity crystals were generally 6 to 8 inches long. Typical samples are illustrated in Figures 1 and 2.

B. Composition

The chemical composition of all of the crystals placed in the program are listed in Table V. In addition to unalloyed tungsten, crystals with the following additions were also prepared: Nb, Zr, Ti, Hf, Ir, Ta, TaC, ThO_2, and $K_2SiO_3 + Al_2O_3$. The Zr, Hf and Ir alloys proved to be polycrystalline, and in the case of Zr, a two-phase structure resulted with concentrations of Zr as low as 0.3%. The additions of the metallic elements were made at approximately one atomic percent. The compounds were added in larger quantities since during the growth process they tended to volatilize rapidly.

The carbon and oxygen contents were obtained by a special variation of the fusion-conductometric method [21] which permitted measurement at very low concentration. The trace metallic impurity results are based on emission spectroscopy and are only accurate to within an order of magnitude, e.g., -3 indicates a range 10^{-3} to 10^{-2}% (.001 to .01%). The alloying elements were obtained by standard wet chemical analyses including an ion exchange column.

C. Working Techniques

Conventional hot-working facilities were used successfully to swage, forge or roll the single crystals. Included was the preheating of the dies by first swaging a piece of mild steel. The axial temperature rise of the dies was quite small. This is contrasted with the rather complex methods required for polycrystalline tungstens. The surface was prepared prior to working to minimize fracture and cleavage since it had been found that the removal of a small amount of tungsten improves its ductility and fabricability.[17][22]

Working was done either one of two ways. The first method consisted of centerless grinding followed by pickling in a 60-40 HNO_3-HF bath. After this treatment the crystals were then swaged. The other method was to pickle in the 60-40 HNO_3-HF bath, then forge to a

flat (about 50% the original diameter), pickle again and then roll. These procedures were determined experimentally as the first part of the program.

The working schedule used was to preheat the specimens in a tube or muffle filled with argon followed by one pass in the die, forge or mill followed by a reheating to 1200°C. Analytical results indicate very little contamination of the material during working. (See Table V.) The swaging went by 1/16 inch increments above 1/2 inch in diameter and by 1/32 inch increments below 1/2 inch in diameter. Most of the material was kept intact after a diameter of 3/8 inch was reached as this was the desired size for tensile specimens. A few sections of crystals were swaged down to .200 inch. Forging took the crystals down to about 1/2 their original diameters. About 3 reheats for the smaller diameter crystals (1/2 inch) and about 7 reheats for the large diameter crystals (1 inch) were needed. Hot rolling was done by increasing the draft .030 inch after each pass until the thickness of the sheet was about .040 inch. At this thickness the rolls were almost jammed. The thinnest sheet produced to date was about .031 inch thick.

During the working operations, various specimens or samples were removed from the crystals. A sample was cut from each crystal after completing a swaging die. Hardness readings were taken on these samples and in some cases the recrystallization temperature was determined. Samples were also removed after forging and various times during the rolling process. For hot hardness readings, specimens were removed after forging or after a few rolling passes. This was because of the requirements on specimen size in the hot hardness testing apparatus.

With the exception of a few specimens described elsewhere, all the crystals were either swaged to .375 inch or rolled to .040 inch. This material was then sand or metal-blasted to remove the oxide coating, sectioned, and then pickled.

D. Metallography

The structure of the as-grown single crystals can be seen in Figures 3-8. Normally, crystals exhibit a cross section as shown in Fig. 3. The boundaries between the "pie slices" are termed lineage here, if the amount of misorientation is less than about 5 degrees.

The misorientation is determined by the crystallographic angle between the normals of the exposed surfaces as determined optically or by means of a back reflection Laue photo. This method is acceptable for misorientations >1 degree; however, for smaller amounts of misorientation a microfocus X-ray tube is required and is currently being used. An example of the type of pattern obtained is shown in Figure 9, with a short explanation on the facing page.

Reflected X-rays can be used to produce images of crystals. This fact was proposed by Garrett [5] and utilized by Schultz [23] to examine the surface of single crystals. Using white radiation and a good point source of X-rays an image of a surface is reproduced on film. The requirement that the Bragg condition be satisfied is taken care of via the use of white radiation. The image produced is an exact replica of the surface exposed. If one exposes a closed packed plane other planes of the zone will also be reflected so that one obtains a series of images as shown in Figure 9.

With the high resolution possible with X-rays and using a microfocus tube the presence of small misorientations can be detected. Areas will reflect consistently unless there is a slight variation in orientation between adjacent sections. In this case a slight overlap or gap will appear and the angular difference can be calculated from geometrical considerations. Since the distance from the specimen to the film magnifies this angular difference, misorientations of 1 minute of arc can be detected.

The areas of misorientation change slightly as one goes from one end of the boule to the other. This can be seen by Figures 7 and 8 which are cross sections of the opposite ends of the same boule. In this case, the amount of misorientation increases and there is some coarsening of the sub-structure. There is almost always some change in the lineage but the amount and direction of change is not always the same. The reason for this is not fully understood but it is believed to be directly associated with the growth process.

Normal metallographic procedure was followed for all micrographs shown. In the case of the myriad of recrystallization specimens, a few short cuts were taken. The specimens normally were not mounted and were heavily etched after being polished on abrasive paper. While this often left some scratches on the surface, one could easily follow the progress of recrystallization. This was spot checked by going through a detailed diamond polishing procedure. The crystals were normally electrolytically etched in a 2% NaOH solution; however, a few of the alloys seemed to etch quite well in a 1:1 solution of $HF-HNO_3$. A fully recrystallized sample is shown in Figure 10.

E. Sample Preparation

Samples for various tests were sectioned appropriately. For the determination of the ductile-brittle transformation temperature, bend specimens were cut from the .040 inch sheet so that the distance between loading points times the constant cross-sectional area = 0.02 $inch^3$. This was done so that approximately a constant strain rate could be obtained through the elastic region. For tensile specimens, 2-1/4 inch lengths were cut from the .375 inch swaged rod and ground to standard size specimens. Drawings and examples of the tensile specimens prepared are shown in Figures 11 and 12. For hot hardness

specimens, circular (or 2 semi-circular) disks 1-1/2 inch in diameter by 1/16 inch to 1/4 inch thick were cut. For room temperature hardness readings or recrystallization temperature determinations, specimens 1/4 to 1/2 inch long were cut from the swaged rod or sections 1/8 inch square or larger were cut from the rolled sheet.

In addition, all of the material was flash pickled with 60-40 HNO_3-HF to improve the surface. Except for the recrystallization temperature specimens and some of the bend-transition specimens very little of the material was removed. In the case of the bend-transition temperature, surface removal was found to have a pronounced effect. While surface condition would probably have an effect on room temperature tensile specimens, this was not investigated for the high temperature tensile tests employed.

EXPERIMENTAL RESULTS

A. Chemical Composition

The only interstitials analyzed were carbon and oxygen. While Table V lists the contents of all the crystals, a typical analysis is shown below based on PE (an unalloyed crystal of [111] orientation).

	Starting Powder	Crystal After Growing	Rod After Swaging	Sheet After Rolling
Carbon	70	10	4	5
Oxygen	400	24	10	32

This is a rather typical crystal since the carbon and oxygen contents in the as-grown crystal are both on the order of 10 to 30 ppm. After working the carbon content drops appreciably while the oxygen content remains about the same. The oxygen content varies randomly both axially and radially and the method of sample preparation was found to be quite critical.

Alloying elements with the exception of Ti appeared to have close to the desired amount present. There also seemed to be a fairly homogeneous distribution of the alloying element; however, the seed end analyzed slightly higher in the majority of cases. With the addition of compounds TaC, ThO_2 and $K_2SiO_3 + Al_2O_3$ only the TaC remained in a large enough percentage to be determined accurately. In the case of a 2% addition of ThO_2 to the starting powder only 0.015% Th was found in the final crystal. In all cases the compound additions permitted the growth of single crystals. There was no significant change in the concentration of an alloying agent as a result of working.

B. Fabrication

The working of all the crystals in the program are described in Tables VI and VII. If fracture occurred during working, it was relatively simple to determine if failure was caused by cleavage or boundaries. The difference is illustrated in Figures 13 and 14. If the material had a high degree of misorientation or a second phase, it tended to fracture along boundaries. If the material became too cool during working or if there were stress raisers present, the material tended to cleave. Examples of the sheet and rod obtained are shown in Figure 15.

Most of the fabricating was done at approximately the same furnace temperature (1200°C). At this temperature three items seemed to have the most pronounced effect upon fabricability: chemical composition, amount of misorientation across boundaries, and orientation.

Chemical composition seemed to have an important effect, especially in the case of alloys. Columbium in the presence of over 50 ppm carbon and Hf additions produced material which was brittle. Generally, the effect of alloying elements was to increase the amount of misorientation. This was even more pronounced in the larger diameter crystals since in all four cases of Nb addition the material could not be forged or swaged. An addition of 1% Zr or Ir produced extremely brittle polycrystalline material which could not be forged. Titanium additions had the least effect of this type and large diameter crystals of this alloy forged, rolled and swaged comparatively easily. This might be related to the lower per cent retention of Ti. Since the interstitial contents were all fairly uniform no correlations can be drawn on the influences of those elements. However, when 100 ppm carbon was present with Ta or TaC the crystals swaged easily.

The amount of misorientation is also quite critical. This is anticipated in view of the known impossibility of fabricating polycrystalline tungsten under these conditions. While this was not extensively studied, the only unalloyed materials which fractured were those with misorientations greater than 5°.

The effect of orientation on fabrication is not yet completely clear. This is particularly true in the case of forging and rolling. One of the problems was that only the rolling direction was taken into account and forging is a non-uniform type of deformation. One remedy would be to grind flats onto the cylinders rather than forge them. Of the limited number of crystals swaged, the [110] orientation had the greatest tendency to break intergranularly and the [100] swaged easiest. Unfortunately, the [100] had a grave tendency to cleave during the centerless grinding operation. Two of the four [100] crystals prepared failed during this operation. This is probably due to the (100) plane being the cleavage plane.

C. Mechanical Property Data

Specimens for tensile tests were ground from the rod swaged to 0.375" and from the sheet rolled to 0.040". All of the tensile test data is listed in Tables VIII to X. In attempts to obtain machinability data, a number of the specimens were lost; consequently, only the radii were machined and then a diamond tool was employed. After preparation the test specimens were flash pickled. The final shape of the specimens is shown in Figure 12.

The single crystals compared quite favorably with the base line polycrystalline specimen X-1 at all of the temperatures examined. The [111] orientation appears to have the highest strength among the unalloyed materials. Alloying additions of Nb, Ta, and Ti had greater tensile strengths than X-1 which was a section of preswaged commercial rod swaged from 0.500" to 0.375" in the same apparatus used for the single crystals. The ThO_2 doped crystals also had high tensile strengths.

Hot hardness specimens were also prepared and measurements taken. The results obtained are listed in Table XI. Only a few of the crystals were tested in this manner. The readings are a good indication of the high temperature strength.[24]

Room temperature hardness values have been obtained on swaged material at various degrees of reduction. The trend of work hardening is shown in Figure 16. Some specimens have also been obtained from the material which was forged and rolled. While hardness values are not as complete for this method of fabrication, the trend seems to be the same. Work hardening is rapid in the early stages of reduction, followed by a relative insensitivity to further work. A sample data sheet is shown in Table XII.

Ductile-brittle transformation temperatures were determined by means of a bend test. Essentially three point loading was used. A 0.040 inch sheet was bent 90 degrees around a 3-T (3 times the thickness) radius. The transition temperatures were determined on a go no-go basis and are listed in Table XIII. A sample data sheet for two of the specimens is shown in Table XIV. The small diameter crystals were all forged to about 0.250 inch from about 0.500 inch and then rolled to 0.040 inch. Crystals of larger diameter were forged to about 0.450 inch from about 1.050 inch and then rolled or cross rolled (DC) to 0.040 inch. The bending apparatus is adjusted to a load rate of 15 pounds/minute. The specimen is prepared and mounted so that the strain rate is also approximately constant through the elastic region. Temperatures reported are for as-worked sheet followed by flash pickling.

Some qualitative results on the effect of surface removal were obtained. Those specimens which were only sand blasted had a transition temperature about 300°C. With less than 0.001" removed from the surface by pickling in 60-40 HNO_3-HF the temperature drops to about 200-250°C. The lower value is obtained from [100] and [210] orientations.

However, with 0.003" removed from surface the transition temperatures appear to be in the neighborhood of 100°C.

D. Recrystallization Behavior

Recrystallization temperatures based on isochronal anneals have been determined for the worked materials. These temperatures are listed in Table XV. A sample data sheet is shown in Table XVI. The criterion used for recrystallization was 100% of the microstructure composed of recrystallized grains. Hardness readings in Rockwell A were also taken as a check; however, in some cases the material became so brittle that it shattered under the impact of the test. For this reason, it was felt that room temperature hardness readings could not be accurately used.

Results for representative samples, alloyed and unalloyed, are shown graphically in Figures 19a, 19b, 20a and 20b. From these graphs one can see the potential inconsistencies of using hardness readings exclusively. The progress of recrystallization is shown in Figures 17 and 18 for both unalloyed tungsten and alloyed tungsten.

On a number of the specimens the presence of deformation bands are noted. Some examples are shown in Figures 21-23. Since such bands are not always apparent, it is possible that orientation plays an important role in the deformation process and the properties obtained after deformation. This is further illustrated by the difference in recrystallization temperatures listed in Table XV. For example, PHA and PKA were both swaged to about 60% R.A. and PHA (100) had a recrystallization temperature of 1775°C while PKA (110) had a recrystallization temperature of only 1650°C. Although this difference is significant, other orientations e.g., PEB (111) showed about the same recrystallization temperature as PHA and a much higher tensile strength. On this basis and on comparing the tensile strengths at 1000°C of the swaged crystals with their recrystallization temperature a direct correlation is not evident. However, all of the alloys with high tensile strength at 1000°C also had high recrystallization temperatures. The relationship is shown in Table XVII.

DISCUSSION

A. Chemical Composition

On the basis of the results listed in Table V the purity of the unalloyed crystals ranged between 99.9% and 99.99+% W. The alloys were similar in composition except for the addition element. In some cases (RA-RF) care was taken to produce high purity-high perfection (crystallographically) single crystals. These materials did not have any features highly distinguishable from those prepared in the normal manner. Since the carbon and oxygen contents were consistently quite low and the metallic impurities varied randomly, no effect of trace

elements could be observed. This does not imply that higher purity material will not have different properties and improved fabrication. It does indicate, however, that the ranges 5 - 25 ppm carbon and oxygen and 50 - 500 ppm metallic impurities have essentially the same fabricability and properties.

The effect of alloying agents can be seen by an inspection of the tensile properties and recrystallization temperature. In the few systems studied, Nb, Ta, and Ti definitely impart high temperature strength to the material. Other additions have less desirable effects; for example, Ir and Zr tend to make the material polycrystalline and $K_2SiO_3 + Al_2O_3$ impairs the high temperature strength.

One other interesting feature of the analyses obtained is the loss in carbon during working. While the materials were heated in an argon filled muffle, they were worked in the atmosphere. The oxide coating which forms apparently decarburizes the tungsten to an appreciable extent in much the same manner that steel is decarburized. This is believed to be the only way the carbon could have been lost for the starting material either showed no difference in the radial concentration of carbon or else indicated that the carbon concentration was higher in the interior. With a higher carbon concentration in the interior one would expect an increase in the amount of carbon present after working since the surface is removed. Another factor which points to a skin effect is the slight and random changes in oxygen concentration before and after working.

B. Fabricability

The fact that single crystals of tungsten and its alloys are easily fabricated at 1200°C is pointed out by Tables VI and VII. Of the 65 crystals placed in the program only 18 broke. Of the 18 which fractured the major problem was polycrystallinity or misorientations caused mainly by the alloying agents. The (100) orientation seemed to swage well; however, 2 of the 4 crystals cleaved during centerless grinding. One crystal broke due to poor surface preparation which is also critical.

The biggest percentage of failures occur during the swaging operation. The massive swaging dies cool the material and the temperature drops rapidly. One observes this readily by noticing the rapid change in color. In the forging and rolling operations the tungsten is not in continuous contact with colder materials and consequently does not cool as rapidly. With this in mind, it appears that by keeping the swaging dies hot, the number of failures for good quality single crystals would be negligible.

C. Mechanical Properties

Tungsten's properties are difficult to correlate due to the importance of effects such as composition, strain rate, and surface preparation. The importance of surface preparation was evident early in the program when its pronounced effect upon fabricability was ascertained. Unless care was taken in the preparation of the material for working, there was little probability of successfully swaging or forging the crystals. This effect was also pointed out during the ductile-brittle transition temperature tests where proper preparation of the surface lowered the transition temperature by as much as 200°C.

Mechanical properties of single crystal tungsten and its alloys show it to be comparable to polycrystalline material. Tensile strengths when compared with X-1, the polycrystalline rod added for comparison, or with values in the literature are similar. There is, of course, an effect of orientation, with the (100) having the lowest strength. This is in rough agreement with the work of Ferriss et. al., whose specimens were not previously worked.[25]

Ductile-brittle transition temperatures also indicate that single crystals after working performed in a manner similar to tungsten prepared by other processes. Although single crystals of tungsten have been reported as ductile at room temperature[18][27][25][26], these were not previously worked to any appreciable extent. The transition temperatures reported have ranged from 150-450°C with a high purity material reported to have a transition temperature close to 200°C[10]. Some of these temperatures have been based upon tensile tests and consequently there is no direct correlation with the bend transition test used here; however, the relative ductility should remain unchanged. For example, Atkinson[10] notes a decrease in transition temperature of about 100°C as the purity of the material increases, while Stephens[17] reports a decrease in the ductile-brittle transition temperature of about 150°C as the outer surface is removed. Thus, a lowering of the transition temperature of worked single crystals of high purity to about 100°C upon appropriate surface treatment appears consistent.

The validity of hot hardness tests as a useful measure of high temperature strength has already been established.[24] Such measurements on worked crystal produce results which correlate qualitatively with tensile strength and recrystallization temperature (Table XI). It should be noted that differences in recrystallization temperatures, tensile tests, and hot hardness values were not large enough for a quantitative evaluation.

Room temperature hardness data were less useful because of the brittleness of the material below its transition temperature. This produced considerable scatter in such data. Table XII illustrates a system which was fairly well behaved.

D. Recrystallization Studies

Using microstructure and 1/2 hour isochronal heats, recrystallization temperatures were determined. These ranged from about 1500 to 2200°C depending upon the alloy and amount of reduction. Since the concept of recrystallization and recrystallization temperature varies throughout the literature*[28], it was decided to use a comparatively simple method (metallographic observation), a short time (1/2 hour) which would permit the entire specimen to arrive at temperature and yet increase the temperature spread between specimens, and a method which was easily reproducible. The purpose of determining the recrystallization temperature was to affect a correlation between high temperature strength and microstructure. Furthermore, recrystallized grain structure has been found to have a pronounced effect upon the creep resistance of tungsten at least in wire form.[29]

The correlation of recrystallization temperature with tensile strength is shown in Table XVII. By the method of least squares an equation for the recrystallization temperature of [111] unalloyed tungsten was derived.

$$y \text{ (Recrystallization Temp.)} = a + b \times (\% \text{ Reduction in area})$$

where $a = 1885°C$ and $b = -3°/\%$ Reduction in area

This equation yielded results within 100°C for all of the unalloyed crystals between 20 and 80% R.A. If one assumes the same sort of relationship for the alloyed crystals, with a change only in the constant a the difference in the recrystallization temperature for the reduction in area employed (45-62%) is almost within the experimental error of $\pm$ 35°C.

It should be noted that all of the materials which recrystallize at a low temperature also have quite low tensile strengths. Thus, recrystallization temperature could be utilized to eliminate studies of alloy systems which are not promising from the strength point of view.

E. Future Work

Additional effort should be expended to obtain retardant recrystallization textures such as have been found for iron alloys.[31] Other techniques could also be employed to retain the single crystal

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* In all cases it is a thermally activated stress relieving phenomena and the temperature where this phenomena starts, stops, or is 50% complete after a given length of time.

nature of the starting ingot. Some ideas along this line include intermediate heat treatments for 100% recovery with no recrystallization (recrystallization in. situ.), hot working via large reductions a material with a low recrystallization temperature, and starting with a number of small single crystals grown in a configuration close to the final desired dimension so only a small amount of cold work has to be employed.

Additional work would also be profitably employed on other alloying combinations. This includes more concentrated additions of Ti, Ta, and Nb, which indicated good high temperature properties in dilute form, and other additions such as Re which have been studied extensively in polycrystalline form.[15]

Further extensive studies of surface preparation conducted in a quantitative manner is also desirable.

CONCLUSIONS

1. Lower temperatures and standard mill equipment can be utilized for fabrication of single crystals of unalloyed tungsten and several dilute solid solutions.

2. The arc Verneuil method of consolidation produces material of reasonably high purity. This is important not only from a fabrication standpoint but also because it permits preparation of consistent materials which are low in interstitial content.

3. The addition of small concentrations of alloying elements produces material with good high temperature strengths.

4. The strength and ductility of worked single crystal tungsten compares favorably with polycrystalline tungsten of similar composition produced by other methods. For worked specimens with a fair amount of surface removed the bend transition temperature is close to 100°C.

5. The results obtained support other observations that removal of contaminated or micro-cracked surface layers enhances the ductility and fabricability of tungsten and its alloys.

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TABLE I

SWAGING TESTS WITH SINGLE CRYSTAL TUNGSTEN
WITHOUT PRIOR ANNEAL*

Orientation	Temperature	Observation
(110)	600°C(1110°F)	Broke on first pass
(110)	700°C(1290°F)	15.7% partial reduction before breaking
(100)	700°C(1290°F)	38.5% partial reduction before breaking
(111)	700°C(1290°F)	35.1% partial reduction before breaking
(100)	1000°C(1830°F)	55% reduction, swaged easily
(111)	1000°C(1830°F)	37% reduction, swaged easily
(210)	1000°C(1830°F)	51% reduction, swaged easily

* Powder metallurgy W requires about 1500°C (2730°F) while arc-melted W requires about 1700°C (3090°F) to swage. The swaging temperature of arc-melted W after impact extrusion can be lowered to 1450°C (2640°F).

TABLE II

SWAGING TESTS ON SINGLE CRYSTAL TUNGSTEN
AFTER STRESS RELIEF AT 1700°C FOR ONE HOUR

Orientation	Temperature	Observation
(100)	900°C(1650°F)	Swaged easily
(100)	~700°C(1290°F)	Swaged easily

TABLE III

SOME PROPERTIES OF TUNGSTEN PREPARED BY VARIOUS METHODS

Property	*Linde Single Crystal[30]	Powder Met.	Zone-Refined Single Crystals	Arc Melted
U.T.S. at 150°C	118,300	124,800[8]	16,000 - 132,000***[24]	122,200[8]
Y.S. at 150°C	105,100	123,800		119,100
Red Area at 150°C	6.0%	1.2	>10%[25] 0-16%[10]	1.9
U.T.S. at 1200°C	35,000	25,000[10]		55,000[30]
Y.S. at 1200°C	34,000	12,000		52,000
Elong. at 1200°C	10%			13.0%
U.T.S. at 1650°C	10,000	13,000[30]		14,000[30]
Y.S. at 1650°C	10,000	7,500		9,800
Elong. at 1650°C	**	32%		42%

* Approximately 1/2 inch diameter arc-Verneuil grown crystal. Tensile sample surface mechanically ground.

** Chisel-like deformation

*** These were tested at 100°C and were of various orientations. The highest strength recorded is for a specimen with a [111] axis.

All strengths reported are in psi.

TABLE IV

MECHANICAL PROPERTY TESTS OF W ALLOYS--POLYCRYSTALLINE

1650°C (3000°F) Strain Rate 0.02 min.$^{-1}$

	Yield 0.2% Offset psi	Ultimate Tensile Strength psi	Elongation %	Reduction Area %
Arc Melted W	9,800	14,000	41.8	98
Powder Metallurgy W	7,500	13,400	32.0	40.5
W + 0.005% Ti	13,700	17,100	36.0	98
W + 0.12% Zr	40,800	47,700	16.0	38.9
W + 0.57% Nb	50,000	60,600	20.2	81.7
W + 0.88% Nb	45,000	46,100	15.4	84.5

TABLE V

CHEMICAL ANALYSES OF TUNGSTEN AND TUNGSTEN ALLOYS

Explanation of Terms:

-	- None determined (Analysis was tried and the amount present was below the limit of detectibility.)
N.A.	- Not analyzed
A.R.	- As rolled (the crystal was first forged and then rolled. The analyses was determined after working.)
A.S.	- As swaged (the crystal was swaged and the analysis was determined after working.)
Seed End	- The section of the crystal which was grown first.
Cap End	- The section of the crystal which was grown last.
[hkl]	- The Miller indicies of the growth direction of the axis of the crystal.
-4,-3,-2	- The amount of an element present as determined by emission spectroscopy. Accuracy is within an order of magnitude, e.g., -3 stands for 10^{-3} to 10^{-2}%.
A.G.	- As grown (the crystal was analyzed after growth but prior to any mechanical work.)

All percents given are weight %. All percents are followed by a percent sign. All other data is in parts per million.

The limits of detection of Fe and Si is approximately 0.001% (10 ppm) and of Mo is approximately 0.005% (50 ppm) in a tungsten matrix.

Carbon and oxygen contents are accurate to $\pm$ 10% above 20 ppm and accurate to $\pm$ 100% below 5 ppm.

TABLE V

Crystal; Axis; Condition	Nominal Composition	C	O	Fe	Si	Mo	Ni	Ca	Cu	Mg	Al	Cr	Alloying Agent
Starting Powder	Unalloyed for "P" Series	70	400	-2	-2	-3	-2	-3	-4	-3	-	-3	-
PA [111] AG	Unalloyed	24	22	-3	-3	-2	-3	-	-	-	-	-	-
PB [111] AG	"	30	20	-	-	-2	-3	-	-	-	-	-	-
PB [111] AS	"	7	10	-2	-	-3	-	-	-	-	-	-	-
PC [111] AG	"	17	23	-3	-3	-	-	-	-	-	-	-	-
PD [100] AG	"	11	2	-	-	-2	-3	-	-	-	-	-3	-
PE [111] AG	"	10	24	-	-	-2	-3	-	-	-	-	-	-
PE [111] AS	"	4	10	-	-3	-	-	-	-	-	-	-	-
PE [111] AR	"	5	32	-3	-	-	-	-	-	-	-	-	-
PF [210] AG	"	8	6	-	-	-2	-3	-	-	-	-	-	-
PG [111] AG	"	24	12	-3	-3	-	-	-	-	-	-	-	-
PH [100] AG	"	41	10	-	-	-	-	-	-	-	-	-	-

-Continued-

TABLE V Cont'd.

Crystal; Axis; Condition	Nominal Composition	C	O	Fe	Si	Mo	Ni	Ca	Nb	Cu	Mg	Al	Cr	Alloying Agent
PH [100] AS	Unalloyed	2	5	-	-	-	-	-	-	-	-	-	-	-
PI [100] AG	"	22	2	-3	-	-	-	-	-	-	-	-3	-3	-
PI [100] AR	"	2	48	-2	-3	-2	-	-	-	-4	-	-	-3	-
PJ [110] AG	"	4	4	-3	-	-3	-	-	-	-	-	-	-	-
PK [110] AG	"	16	14	-	-	-	-	-	-	-	-	-	-	-
PK [110] AS	"	9	8	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	-
PK [110] AS	"	6	26	-	-3	-	-	-	-	-	-	-	-	-
PL [210] AG	"	18	14	-	-	-3	-	-	-	-	-	-	-	-
PM [210] AG	"	22	13	-	-	-	-	-	-	-	-	-	-	-
PM [210] AR	"	5	15	-	-3	-	-	-	-	-	-	-	-3	-
PN [112] AG	"	21	8	-3	-	-	-	-	-	-	-	-	-	-
DA [111] AG Seed End	Unalloyed Large Dia.	9	4	-	-	-2	-	-	-	-4	-	-	-	-

-Continued-

TABLE V Cont'd.

Crystal; Axis; Condition	Nominal Composition	C	O	Fe	Si	Mo	Ni	Nb	Cu	Mg	Al	Cr	Alloying Agent
DL [111] AG	3% ThO_2	10	19	-	-	-2	-	-3	-	-4	-	-	Th .015%
DL [111] AR	3% ThO_2	4	10	-	-	-	-	-	-	-	-	-	Th <.01%
DM [111] AG	2% K_2SiO_3+Al_2O_3	33	15	-	-3	-2	-	-	-	-	-3	-	N.A.
DM [111] AS	2% K_2SiO_3+Al_2O_3	6	6	-	-3	-	-	-	-	-	-3	-	N.A.
DN [111] AG	2% K_2SiO_3+Al_2O_3	30	9	-3	-3	-2	-	-	-	-	-3	-	N.A.
DO [111] AG Cap	0.38% TaC	108	2	-	-	-	-	-	-	-	-	-	Ta <0.7%
DO [111] AS	0.38% TaC	110	9	-	-	-	-	-	-	-	-	-	Ta 0.31%
DP [111] AG	0.38% TaC	40	13	-3	-	-2	-	-	-	-	-3	-	Ta 0.30%
DQ [111] AG	Large Dia. 1.0% Ti	7	4	-3	-2	-	-3	-	-	-3	-	-	Ti 0.11%
DR [111] AG	Large Dia. 1.0% Ti	7	2	-3	-2	-	-3	-	-	-3	-	-	Ti N.A.
DS [111] AG	Large Dia. 0.5% Nb	4	3	-3	-	-	-	5000	-	-4	-	-3	Nb 0.36%

-Continued-

TABLE V Cont'd.

Crystal; Axis; Condition	Nominal Composition	C	O	Fe	Si	Mo	Ni	Nb	Ca	Cu	Mg	Al	Cr	Alloying Agent
DT [111] AG	Large Dia.	3	7	-	-3	-	-	5000	-	-	-2	-	-	Nb N.A.
DU [111] AG	0.38% TaC	124	6	-3	-3	-2	-2	-	-	-	-4	-	-3	Ta N.A.
DV [111] AR	Unalloyed Large Dia.	4	78	-	-3	-	-	-	-	-	-4	-	-	-
DW [111]	0.1% Zr	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	-
DX [111] AS	3.0% ThO_2	5	14	-	-	-	-	-	-	-	-	-	-	Th < .01%
DY [111] AG	Large Dia. 1.0% Ta	2	3	-	-	-	-	-	-	-	-	-	-	Ta 0.88%
DZ [111] AG	Large Dia. 1.0% Ta	3	5	-	-2	-	-	-	-	-	-	-	-	N.A.
Starting Powder	Unalloyed For "D" Series	55	70	-3	-2	-2	-3	-	-4	-4	-4	-3	-	-
Starting Powder	Unalloyed For "R" Series	24	185	-3	-3	-3	-	-	-	-	-4	-	-	-
RA [111] AG	Unalloyed	2	13	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	-
RA [111] AS	"	3	4	-	-	-	-	-	-	-	-	-	-	-
RB [111] AR	"	2	34	-2	-3	-	-	-	-	-	-	-	-	-
RC [111] AG Seed	"	4	5	-	-	-	-3	-	-	-	-	-	-	-

-Continued-

TABLE V Cont'd.

Crystal; Axis; Condition	Nominal Composition	C	O	Fe	Si	Mo	Ni	Nb	Cu	Mg	Al	Cr	Alloying Agent
DB [111] AR Cap End	Unalloyed Large Dia.	6	22	-3	-3	-2	-	-	-4	-	-	-3	-
DC [111] AR	"	5	42	-	-	-	-	-	-	-	-	-	-
DD [111] AG	"	12	5	-3	-	-3	-	-	-	-	-	-	-
DE [111] AG	1.0% Ta	6	6	-	-3	-2	-	-	-4	-4	-	-3	Ta 1.01%
DE [111] AS	1.0% Ta	3	7	-3	-	-	-	-	-	-	-	-	Ta 1.03%
DF [111] AR	1.0% Ta	4	2	-	-	-	-	-	-	-	-	-	Ta N.A.
DG [111] AG	1.0% Ti	10	4	-	-3	-2	-	-2	-	-	-	-	Ti 0.09%
DG [111] AR	1.0% Ti	4	2	-	-	-	-	-	-	-	-	-	Ti 0.14%
DH [111] AG	1.0% Ti	14	11	-	-	-2	-3	-2	-4	-	-2	-	Ti 0.18%
DI [111] AG	1.0% Zr	6	13	-3	-	-	-	-	-	-	-	-	Zr -1
DJ [111] AG	0.3% Zr	12	13	-3	-	-	-	-	-	-	-	-	Zr -1
DK [111] AS	3% ThO_2	3	7	-3	-2	-	-3	-	-	-4	-	-	Th <.01%

-Continued-

TABLE V Cont'd.

Crystal; Axis; Condition	Nominal Composition	C	O	Fe	Si	Mo	Ni	Ca	Cu	Mg	Al	Cr	Other	Alloying Agent
RC [111] AS	Unalloyed	2	3	-3	-3	-	-	-	-	-	-	-	-	-
RD [111] AG Cap	"	34	4	-	-	-2	-	-	-	-	-	-	-	-
RD [111] AR	"	6	15	-2	-	-	-	-	-	-4	-	-	-	-
RE [111] AS	"	3	9	-3	-	-	-	-	-	-	-	-	-	-
RF [111] AR	"	5	17	-3	-	-	-	-	-	-	-	-	-	-
RG [111] AG Seed	1.1% Hf	7	6	-	-3	-	-	-	-	-	-	-	Zr -3	Hf -1
RH [111] AG Cap End	1.1% Hf	4	8	-2	-2	-	-3	-	-	-	-	-	-	Hf -1
RH [111] AS	1.1% Hf	2	3	-2	-2	-	-	-	-	-	-	-	-	Hf -1
RI [111] Ag Seed End	1.0% Ir	6	6	-	-	-	-	-	-	-	-	-	Pt -3	Ir -1
RI [111] AG Cap End	1.0% Ir	7	7	-3	-3	-2	-	-	-	-4	-	-	Pt -3	Ir -1
RK [111] AG Seed End	0.6% Nb	7	5	-	-	-2	-	-	-	-	-	-	-	Nb 0.50%
RK [111] AS	"	9	2	-	-	-2	-	-	-	-	-	-	Al 500	Nb 0.63%

-Continued-

TABLE V Cont'd.

Crystal; Axis; Condition	Nominal Composition	C	O	Fe	Si	Mo	Ni	Cr	Mg	Al	Other	Alloying Agent
RL [111] AG Cap End	0.6% Nb	5	7	-	-	-2	-	-	-	-	-	Nb 0.51%
RL [111] AR	"	8	7	-	-3	-2	-	-	-4	-	-	Nb N.A.
RO [111] AG Cap End	"	9	13	-	-	-	-	-	-	-	-	Nb 0.46%
RP [111] AG Seed End	"	11	5	-	-	-	-	-	-	-	-	Nb 0.54%
RP [111] AS	"	4	21	-	-	-	-	-	-	-	-	Nb N.A.
RR [111] AG Cap End	"	98	7	-	-	-	-	-	-	-	-	Nb 0.43%
RR [111] AG Seed End	"	61	14	-3	-3	-	-	-	-	-	-	Nb 0.63%
RS [111] AG Cap End	"	18	18	-	-3	-	-	-3	-4	-	-	Nb 0.31%
RS [111] AG Seed End	"	19	9	-3	-3	-3	-	-	-4	-	-	Nb 0.61%
RT [111] AG Cap End	"	18	5	-	-	-	-	-	-	-	-	Nb 0.47%
RT [111] AG Seed End	"	9	55	-3	-	-2	-	-	-	-3	-	Nb 0.40%
RT [111] AR	"	3	10	-	-	-	-	-	-	-	-	Nb N.A.

-Continued-

TABLE V Cont'd.

Crystal; Axis; Condition	Nominal Composition	C	O	Fe	Si	Mo	Cr	Mg	Al	Alloying Agent
RU [111] AG Cap End	0.6% Nb	15	4	-	-	-	-	-	-	Nb 0.26%
RU [111] AG Seed End	"	6	4	-	-3	-	-	-4	-	Nb 0.45%
RU [111] AR	"	4	7	-	-	-	-	-	-	Nb 0.63%
RV [111] AG Cap End	"	92	11	-	-	-	-	-	-	Nb 0.41%
RV [111] AG Seed End	"	47	3	-	-	-2	-	-	-	Nb 0.39%
RW [100] AG	"	5	2	-3	-2	-	-	-4	-	Nb N.A.
X3 [111] AG Center	Unalloyed	N.A.	3	N.A.	-	-	-	-	-	-
X3 [111] AG Exterior	"	N.A.	3	N.A.	-	-	-	-	-	-

TABLE VI

THE BEHAVIOR OF TUNGSTEN AND TUNGSTEN ALLOY SINGLE CRYSTALS DURING SWAGING TO ABOUT 50% R.A.

Crystal	Axis	Nominal Composition	Swaged Well	Fractured Slightly at Ends Only	Fractured or Cleaved Completely	Remarks
SMALL DIAMETER						
PAA	111	Unalloyed			X	No surface preparation; large misorientation.
PAB	111	Unalloyed			X	No surface preparation; vacuum annealed at 1800°C for 1 hour, cleaved in half at 20% R.A.
PAC	111	Unalloyed		X		No surface preparation; less misorientation than PAA.
PBA	111	Unalloyed	X			Centerless ground; some surface cracks appeared but were ground out.
PBB	111	Unalloyed	X			Centerless ground; pickled; vacuum annealed at 1800°C for 1 hour.
PBC	111	Unalloyed	X			Centerless ground; pickled, a small section was swaged to 89% R.A.
PCA	111	Unalloyed	X			Pickled.
PCB	111	Unalloyed		X		Pickled; one end fractured due to misorientation.
PCC	111	Unalloyed		X		Pickled; vacuum annealed at 1800°C for 1 hour.

-Continued-

TABLE VI Cont'd.

Crystal	Axis	Nominal Composition	Swaged Well	Fractured Slightly at Ends Only	Fractured or Cleaved Completely	Remarks
SMALL DIAMETER						
PEB	111	Unalloyed	X			All the rest of the crystals on the list have been centerless ground and pickled.
PDB	100	Unalloyed			X	This one cleaved along cracks started during grinding.
PH	100	Unalloyed	X			Swaged easiest.
PFB	210	Unalloyed		X		Slight surface cracking one end large misorientation
PL	210	Unalloyed		X		
PK	110	Unalloyed		X		Cracking believed to be partially caused by orientation.
PN	112	Unalloyed	X			
RA	111	Unalloyed	X			
RC	111	Unalloyed	X			
RE	111	Unalloyed	X			
RH	111	1% Hf			X	This was not a single crystal.
RK	111	.6% Nb	X			Had a very low interstitial content and low misorientation.
RO	111	.6% Nb		X		Believed to be caused by interstitial content.

-Continued-

TABLE VI Cont'd.

Crystal	Axis	Nominal Composition	Swaged Well	Fractured Slightly at Ends Only	Fractured or Cleaved Completely	Remarks
SMALL DIAMETER						
RP	111	.6% Nb		X		Believed to be caused by interstitial content; vacuum annealed at 1800°C for 1 hour, no appreciable difference noted.
RR	111	.6% Nb			X	Believed to be caused by high interstitial content.
RS	111	.6% Nb			X	Believed to be caused by high interstitial content.
DE	111	1% Ta		X		Possible due to misorientation or chemical composition.
DH	111	1% Ti	X			
DI	111	1% Zr			X	This was not a single crystal.
DK	111	2% ThO_2	X			
DM	111	2%(K_2SiO_3+Al_2O_3)	X			
DO	111	0.38% TaC		X		Possibly a combination of misorientation and chemical.
DW	111	0.3% Zr			X	This was not a single crystal.
DX	111	3% ThO_2	X			
DV	111	0.38% TaC			X	Operator error.
RW	100	.6% Nb			X	Cleaved during centerless grinding.
X1	Polycrystalline	Unalloyed		X		Commercial purity swaged tungsten rod added to program for comparison.

-Continued-

TABLE VI Cont'd

Crystal	Axis	Nominal Composition	Swaged Well	Fractured Slightly at Ends Only	Fractured or Cleaved Completely	Remarks
LARGE DIAMETER						
DA	111	Unalloyed			X	Believed to be mainly caused by misorientation; possibly permitted to get too cold (0.945 inch).
DD	111	Unalloyed		X		Swaged to .375 inch from .903 inch.
DV	111	Unalloyed	X			
DQ	111	1% Ti	X			
DSA	111	1% Nb			X	Misorientation.
DSB	111	.6% Nb			X	Misorientation.
DY	111	1% Ta			X	Misorientation.

TABLE VII

THE BEHAVIOR OF VARIOUS TUNGSTEN AND TUNGSTEN ALLOY CRYSTALS DURING FORGING TO ABOUT 1/2 ORIGINAL DIAMETER FOLLOWED BY ROLLING TO .040 INCH

Crystal	Axis	Nominal Composition	Forged Well	Broke During Forging	Rolled Well	Some Tearing During Rolling	Remarks
SMALL DIAMETER							
PEA	111	Unalloyed	X		X		
PG	111	Unalloyed	X	X	X		One section which wasn't pickled broke during forging.
PDA	100	Unalloyed	X		X		
PI	100	Unalloyed	X		X		
PFA	210	Unalloyed	X		X		
PM	210	Unalloyed	X		X		
PJ	110	Unalloyed	X			X	Possibly due to orientation.
RB	111	Unalloyed	X		X		
RD	111	Unalloyed	X		X		
RF	111	Unalloyed	X		X		
RG	111	.6% Hf		X			Polycrystalline
RI	111	.6% Ir		X			Polycrystalline

-Continued-

TABLE VII Cont'd.

Crystal	Axis	Nominal Composition	Forged Well	Broke During Forging	Rolled Well	Some Tearing During Rolling	Remarks
SMALL DIAMETER							
RL	111	.6% Nb	X		X		
RU	111	.6% Nb	X			X	Probably due to high interstitial content.
RV	111	.6% Nb	X			X	Probably due to high interstitial content.
RT	111	.6% Nb	X		X		
DF	111	1% Ta	X		X		
DG	111	1% Ti	X		X		
DJ	111	1% Zr		X			Polycrystalline
DL	111	2% ThO_2	X		X		
DN	111	2% (K_2SiO_3+Al_2O_3)	X		X		
DP	111	0.38% TaC	X			X	Possibly due to chemical composition.
RX	100	.6% Nb	X		X		
X2	110	Unalloyed	X		X		Electron beam zone refined single crystal.
LARGE DIAMETER							
DB	111	Unalloyed	X		X		
DC	111	Unalloyed	X			X	Cross rolled.

-Continued-

TABLE VII Cont'd.

Crystal	Axis	Nominal Composition	Forged Well	Broke During Forging	Rolled Well	Some Tearing During Rolling	Remarks
LARGE DIAMETER							
DR	111	1% Ti	X				
DTA	111	1% Nb		X			Misorientation
DTB	111	.6% Nb		X			Misorientation
DZ	111	1% Ta	X			X	

TABLE VIII

TENSILE PROPERTY DATA AT 1000°C ON SWAGED ROD

Crystal	Axis	Nominal Composition	Y.S. psi	U. T. S. psi	% Elongation 1"
RK	111	0.6% Nb	77,400	77,400	12.7
DE	111	1% Ta	75,100	75,100	8.1
PB	111	Unalloyed	66,700	66,900	7.8
DQ	111	1% Ti	60,700	63,500	10.7
PEB	111	Unalloyed	58,200	61,400	4.7
X-1	Polycrystalline	Unalloyed	55,700	61,000	13.4
DX	111	3% ThO_2	59,500	60,200	10.0
DK	111	2% ThO_2	59,700	59,700	6.0
RA	111	Unalloyed	58,000	58,000	10.5
PN	112	Unalloyed	57,800	57,900	10.6
PK	110	Unalloyed	56,600	56,700	10.5
PL	210	Unalloyed	48,800	49,000	12.6
PH	100	Unalloyed	45,300	45,300	11.6
PFB	210	Unalloyed	44,200	44,200	5.4
DM	111	2%(K_2SiO_3+Al_2O_3)	38,600	38,600	6.0

NOTE: All of the swaged rod had 50-60% reduction in area by swaging.

TABLE IX

TENSILE PROPERTY DATA AT 482°C ON SWAGED ROD

Crystal	Axis	Nominal Composition	Y.S. psi.	U.T.S. psi.	% Elongation 1"
PEB	111	Unalloyed	81,800	86,600	6.0
DQ	111	1% Ti	79,900	84,300	13.8
DX	111	3% ThO_2	82,500	84,000	13.0
X1	Polycrystalline	Unalloyed	71,200	80,500	14.2
RC	111	Unalloyed	69,500	73,100	8.0
RA	111	Unalloyed	69,000	71,200	10.7
PFB	210	Unalloyed	58,200	61,700	7.5
PH	100	Unalloyed	50,700	53,200	15.8

TABLE X

TENSILE PROPERTIES OF TUNGSTEN SHEET ROLLED FROM SINGLE CRYSTALS

Crystal	Nominal Composition	Growth Direction	Testing Temp., °C	Yield Strength in Kpsi	Ultimate Strength in Kpsi	Percent Elongation in 1-inch
DB	Unalloyed	(111)	482	107.0	136.5*	2
			482	105.3	141.0	4
			1000	52.0	81.6	4
			1000	71.1	88.9	6
DV	Unalloyed	(111)	482	72.0	111.5	7
			482	88.9	122.9	6
			1000	47.9	78.3	4
			1000	72.3	97.3	5
			1260	37.6	41.0	6
PEA	Unalloyed	(111)	482	126.1	155.8	5
			482	136.7	148.1	5
			1000	70.2	88.0	5
			1000	71.6	82.3	5
DA	Unalloyed	(111)	482	91.6	135.8	3
			482	52.4	77.9*	2
			1000	50.9	92.7	7
			1000	54.2	84.6	6
RF	Unalloyed	(111)	482	134.9	149.5	3
			482	107.9	139.7	5
			1000	84.6	99.2	6
			1000	71.7	95.3	4
			1260	47.8	66.4	6
PFA	Unalloyed	(210)	482	105.2	149.9	5
			1000	48.1	82.3	5
PDA	Unalloyed	(100)	482	131.2	161.2	9
			1000	6[illegible].3	82.3	7
			1260	26.6	32.7	17
DZ	1% Ta	(111)	482	185.0	218.0*	2
			482	105.0	123.0	4
			1000	78.0	127.0	4
			1000	86.1	125.0	4

* Specimen failed in the tab.

TABLE XI

HARDNESS VALUES VERSUS TEMPERATURE FOR SHEET BAR FABRICATED FROM SINGLE CRYSTAL TUNGSTEN

CRYSTAL Alloying Agent Growth Orientation Hardness Value at -	PM Unalloyed (210)	PE Unalloyed (111)	PF Unalloyed (210)	PG Unalloyed (111)	PD Unalloyed (100)
900°F	150	214	-	182	182
1000°F	147	201	205	172	172
1100°F	140	205	197	163	170
1200°F	133	193	182	165	163
1300°F	131	199	175	159	161
1400°F	110	172	162	150	153
1500°F	110	172	156	153	143
1600°F	98.8	150	145	157	139
1700°F	98.8	145	129	127	123
1800°F	100	145	131	125	114
1900°F	97.4	131	117	116	111
2000°F	90.7	121	100	116	97.4
2100°F	94.6	103	73.4	101	91.0
2200°F	98.8	85.8	69.8	94	75.8
2300°F	88.2	45.9	49.6	85	46.4
2400°F	57.9	39.7	44.6	43	40.9

NOTE: The hardness values given above are in arbitrary units and therefore are of a relative nature.

TABLE XII

SAMPLE DATA SHEET ON ROOM TEMPERATURE HARDNESS

Crystal: DD Orientation: (111)

Composition: Unalloyed large diameter

Procedure: Centerless-ground pickled swaged

Diameter prior to swaging - 0.903 in.

Area prior to swaging - 0.64 square inches

Percent Work	R_A Hardness Along Radius	Average Hardness
0	65.3, 66.9, 66.3	66.2
11.4	68.9, 68.1, 68.5	68.5
32.5	68.0, 68.9, 70.0	69.0
42.0	67.1, 66.9, 68.9	67.6
52.1	72.0, 71.2, 71.1	71.4
65.7	70.9, 70.1, 70.1	70.4
69.8	72.0, 71.9, 71.9	71.9
73.9	71.8, 71.8, 72.1	71.9
77.2	71.8, 71.6, 71.9	71.6
80.4	68.0, 70.0, 68.9	69.0
83.8	71.3, 71.0, 70.9	71.1

TABLE XIII

DUCTILE-BRITTLE TRANSFORMATION TEMPERATURE

Specimen	Axis	Nominal Composition	Transition Temperature °F	Transition Temperature °C	Remarks
NO SURFACE REMOVAL					
PGA	111	Unalloyed	525	274	
PGB	111	Unalloyed	575	302	
FLASH PICKLE					
PGC	111	Unalloyed	425	219	
PI	100	Unalloyed	400	204	
PIX	100	Unalloyed	525	274	Cross rolled; transverse specimen.
PFA	210	Unalloyed	400	204	
X-2	110	Unalloyed	>400	>204	Not a valid test, only two specimens.
PJA	110	Unalloyed	425	219	
PJB	110	Unalloyed	425	219	
PEA	111	Unalloyed	450	232	
PM	210	Unalloyed	475	246	
RB	111	Unalloyed	500	260	
RD	111	Unalloyed	500	260	
PDA	100	Unalloyed	550	288	
DV	111	Unalloyed	550	288	
RF	111	Unalloyed	575	302	
DC*	111	Unalloyed	575	302	Cross rolled; transverse specimen.
DB*	111	Unalloyed	600	316	1600°C 1 hr. prior test.
DB*	111	Unalloyed	500	260	1200°C 1 hr. prior test.
DB*	111	Unalloyed	375	191	
DN	111	$K_2SiO_3+Al_2O_3$	375	191	
RY	100	.6% Nb	375	191	
DR*	111	1% Ti	450	232	
DG	111	1% Ti	475	246	
RL	111	.6% Nb	500	260	
RU	111	.6% Nb	550	288	
RT	111	.6% Nb	550	288	
RV	111	.6% Nb	550	288	
DP	111	0.38% Ta	550	288	
DF	111	1% Ta	525	274	
DL	111	1% ThO_2	575	302	
LARGE AMOUNT OF SURFACE REMOVED (0.003")					
DZ*	111	1% Ta	200	93	
DB*	111	Unalloyed	200	93	

* Large diameter crystals

NOTE: Transition temperatures were determined in terms of °F and are accurate to approximately ± 13°.

TABLE XIV

DATA SHEET FOR DETERMINING TRANSITION TEMPERATURE

Temperature °F	Unalloyed Crystal PI [100] Flash Pickled Test Results*	Unalloyed Crystal DB [111] .003" Removed From Surface Test Results*
Room Temp.		X
150		X,0
200		0,0 - Transition Temperature
250	0,X,X,X	X,0
300	0,0,0,+,X	0
350	X,X,X	
400	0,X - Transition Temperature	
450	0,0	
500	0,0	

* X - Specimen fractured

0 - Specimen bent with no cracking

\+ - Specimen bent with some cracking

The reason for some of the crystals bending at 300°F is probably caused by non-uniform surface preparation. Chemical analyses were obtained on a specimen which bent at 300°F and one which fractured at 350°F. No difference was observed. It seems logical to conclude a surface effect causes the scatter.

TABLE XV

RECRYSTALLIZATION TEMPERATURES OF WORKED SINGLE CRYSTALS

Crystal	Axis	Nominal Composition	Type of Working	Recrystallization Temperature °C ± 35°
RO	111	.6% Nb	47% R.A. Swaging	2125
RP	111	.6% Nb	49% R.A. Swaging	2075
PKB	110	Unalloyed	10% R.A. Swaging	2025
DX	111	3% ThO_2	56% R.A. Swaging	1925
PAB	111	Unalloyed	20% R.A. Swaging	1900
DK	111	2% ThO_2	52% R.A. Swaging	1900
DO	111	0.38% TaC	50% R.A. Swaging	1900
RK	111	.6% Nb	51% R.A. Swaging	1900
DE	111	1% Ta	55% R.A. Swaging	1825
RC	111	Unalloyed	50% R.A. Swaging	1800
DH	111	1% Ti	52% R.A. Swaging	1775
X1	Polycrystalline	Unalloyed	50% R.A. Swaging	1775-1800
PHA	100	Unalloyed	60% R.A. Swaging	1775
PBA	111	Unalloyed	56% R.A. Swaging	1750
PCA	111	Unalloyed	60% R.A. Swaging	1750
DM	111	$K_2SiO_3+Al_2O_3$	55% R.A. Swaging	1725
DQ*	111	1% Ti	85% R.A. Swaging	1700
PFB	210	Unalloyed	63% R.A. Swaging	1700
PEB	111	Unalloyed	62% R.A. Swaging	1700
PBC	111	Unalloyed	56% R.A. Swaging	1700
PBB	111	Unalloyed	58% R.A. Swaging	1700
PAC	111	Unalloyed	62% R.A. Swaging	1700

* Large diameter

-Continued-

TABLE XV Cont'd

Crystal	Axis	Nominal Composition	Type of Working	Recrystallization Temperature °C ± 35°
RE	111	Unalloyed	60% R.A. Swaging	1675
PL	210	Unalloyed	61% R.A. Swaging	1675
DD*	111	Unalloyed	78% R.A. Swaging	1650
PKA	110	Unalloyed	60% R.A. Swaging	1650
PNB	112	Unalloyed	62% R.A. Swaging	1625
RA	111	Unalloyed	56% R.A. Swaging	1575
RH	111	1% Hf	20% R.A. Swaging	1525
PGC	111	Unalloyed	As forged	1650**
RU-1	111	.6% Nb	Rolled to .045 in.	2000
RU-2	111	.6% Nb	Rolled to .063 in.	2000
DG	111	1% Ti	Rolled to .042 in.	1950
RV	111	.6% Nb	Rolled to .044 in.	1925
RX	100	.6% Nb	Rolled to .043 in.	1900
RT	111	.6% Nb	Rolled to .061 in.	1875
RD	111	Unalloyed	Rolled to .043 in.	1875
DF	111	1% Ta	Rolled to .042 in.	1850
RL	111	.6% Nb	Rolled to .044 in.	1850
DP	111	0.38% TaC	Rolled to .043 in.	1825
PFA	210	Unalloyed	Rolled to .043 in.	1775
PMA-1	210	Unalloyed	Rolled to .120 in.	1775
DN	111	$K_2SiO_3+Al_2O_3$	Rolled to .042 in.	1750
PGB	111	Unalloyed	Rolled to .041 in.	1750

* Large diameter

** This value seems low but it was rechecked by an additional anneal. Perhaps during hot-rolling some stress is relieved.

-Continued-

TABLE XV Cont'd.

Crystal	Axis	Nominal Composition	Type of Working	Recrystallization Temperature °C ± 35°
PGA	111	Unalloyed	Rolled to .042 in.	1750
PDA	100	Unalloyed	Rolled to .043 in.	1750
PEA	111	Unalloyed	Rolled to .040 in.	1725
PIx	100	Unalloyed	Cross Rolled to .048 in.	1725
DBA	111	Unalloyed	Rolled to .046 in.	1725
DBB*	111	Unalloyed	Rolled to .195 in.	1725
DC*	111	Unalloyed	Cross Rolled to .045 in.	1750
DZ*	111	1% Ta	Rolled to .045 in.	1775
RB	111	Unalloyed	Rolled to .042 in.	1725
RF	111	Unalloyed	Rolled to .042 in.	1725
DL	111	2% ThO_2	Rolled to .042 in.	1725
PIA	100	Unalloyed	Rolled to .056 in.	1700
PJA	110	Unalloyed	Rolled to .040 in.	1675
PMB-1	210	Unalloyed	Rolled to .032 in.	1675
DR*	111	1% Ti	Rolled to .042 in.	1650
DVC	111	Unalloyed	Rolled to .040 in.	1650
X-2	110	Zone refined single crystal	Rolled to .040 in.	1650
PMA-2	210	Unalloyed	Rolled to .046 in.	1700
PJB	110	Unalloyed	Rolled to .041 in.	1625

* Large diameter

TABLE XVI

SAMPLE DATA SHEET USED FOR RECRYSTALLIZATION TEMPERATURES

Temperature °C	PMA-1 [210] Unalloyed Rolled to .120 inch Hardness R_A	Micro*	DN [111] $K_2SiO_2+Al_2O_3$ Rolled to .042 inch Hardness R_A	Micro*	RU-1 [111]1% Nb Rolled to .045 inch Hardness R_A	Micro*
Room Temperature	68.6	0	71.6	0	71.7	0
1250	65.1	25	66.4	5		
1350	61.7	40	63.1	60	69.5	0
1500	60.9	60	63.4	60	66.3	20
1620	61.2	75	60.9	80	63.6	30
1700	60.9	85	61.7	95		
1775	61.2	100	60.0	100	64.1	80
1850					63.7	85
1975					63.0	98-100
2100					62.7	100
Recrystallization Temperature	1775		1750		2000	

* Micro = percent recrystallized by microscopic observation.

TABLE XVII

TENSILE STRENGTHS VS. RECRYSTALLIZATION TEMPERATURE

Specimen	Axis	% R.A.	Nominal Composition	Recrystallization Temp. °C	UTS 1000°C psi
RK	111	51	0.6% Nb	1900	77,400
DE	111	55	1% Ta	1825	75,100
PBB	111	58	Unalloyed	1700	66,900
DQ	111	86	1% Ti	1700	63,500
DX	111	58	3% ThO_2	1875	62,800
PEB	111	62	Unalloyed	1700	61,400
X-1	Polycrystalline	50	Unalloyed	1775	61,000
DK	111	52	2% ThO_2	1900	59,700
RA	111	56	Unalloyed	1575	58,000
PN	112	62	Unalloyed	1625	57,900
PK	110	60	Unalloyed	1650	56,700
PL	210	61	Unalloyed	1675	49,000
PH	100	60	Unalloyed	1775	45,300
PF	210	63	Unalloyed	1700	44,200
DM	111	55	2%(K_2SiO_3+Al_2O_3)	1725	38,600

Fig. 1 - Single crystal of tungsten - 3/4 inch in diameter and 12 inches long. Normal size.

Fig. 2 - Large diameter unalloyed tungsten crystal DC [111].

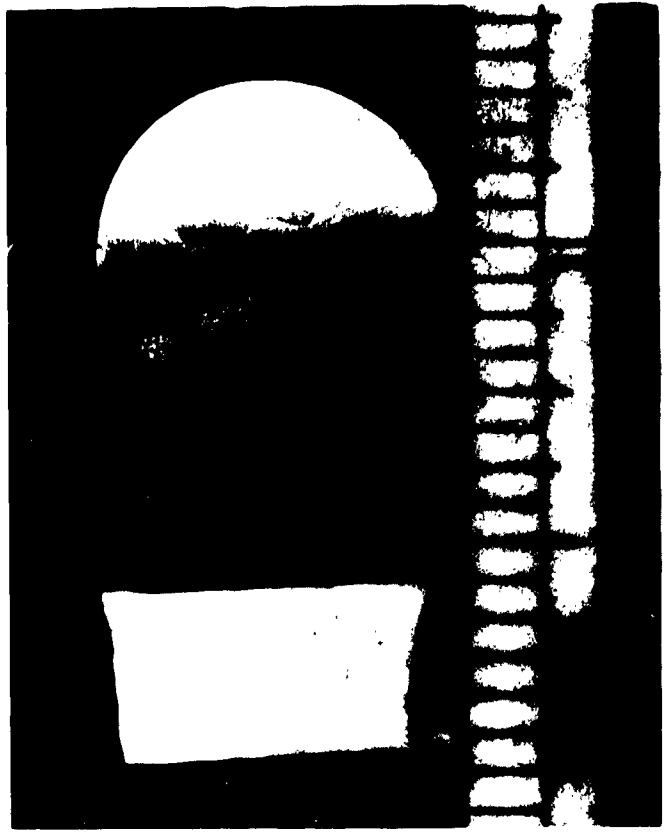


Fig. 3 - Transverse & longitudinal sections of PA, an unalloyed tungsten crystal with an [111] axis. The maximum misorientation was about 4-1/2°.

100X

Fig. 4 - PC [111], an unalloyed tungsten crystal showing the subgrain boundaries & etch pyramids.

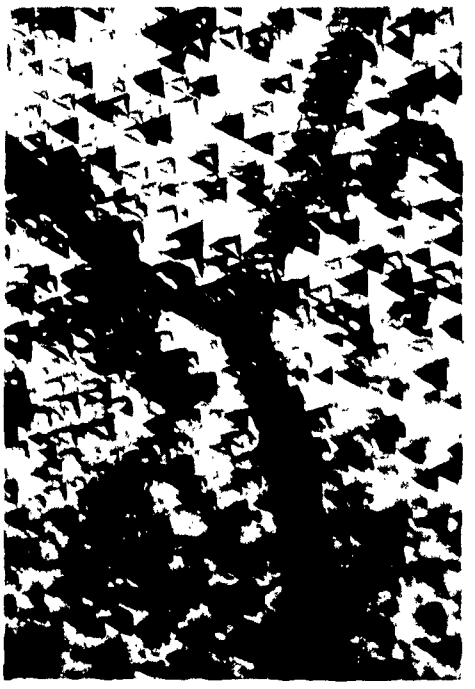


Fig. 5 - PC[111], an unalloyed tungsten crystal showing the triangular etch pyramids characteristic of the (111). 750X

Fig. 6 - PA[111], an unalloyed tungsten crystal shown in Fig. 3. This is the longitudinal section showing the shingle-like structure characteristic of (h,k,0). Note the change in etching at the grain boundary. 750X

3X

Fig. 7 - Seed end of large diameter unalloyed tungsten crystal DB [111]. Note the large number of subgrains and very slight difference in orientation.

EXPLANATION OF FIGURE 9

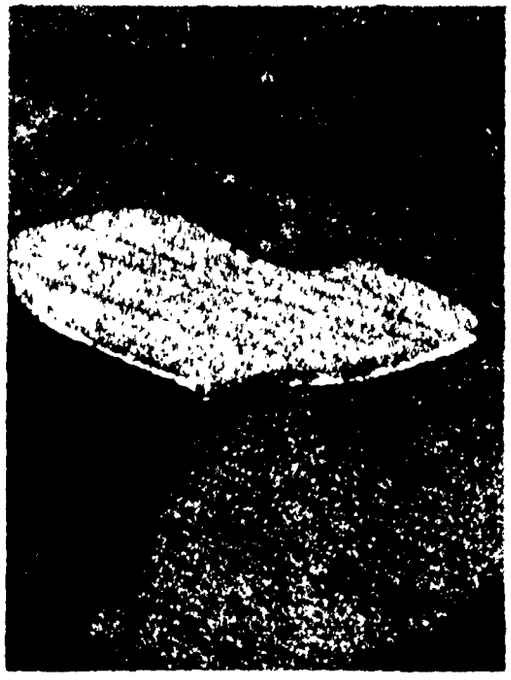
Figure 9 was obtained by using a microfocus tube with a camera similar to that described by Wei [32]. Using unfiltered tungsten radiation, the (100) was lined up parallel to the camera axis. The surface plane of the crystal was 3° from being a (100). The angle between the (100) and the incident beam of X-rays was about 22° for (200) diffraction. The (200) image is located in the upper right central portion of the photograph. Other images are from less closely packed planes in the (100) zone. The other strong image (left center) is from the (420). This pattern was obtained after a 1/2 hour exposure using 35 kilovolts and 350 microamps.

Misorientations based upon the (200) image are approximately 25' of arc for the small section divorced from the top part of the image and approximately 6' of arc for the widest misorientation to the lower right. The heavy white lines are the characteristic L radiation of tungsten. When observed visually there was no misorientation discernable except for the small top section.

3X

Fig. 8 - Cap end of large diameter unalloyed tungsten crystal DA [111]. Note the fewer (comparatively) subgrains and larger amounts of misorientation.

Fig. 9 - Photograph of images obtained from the x-ray microfocus tube. An explanation is included on facing page.



100X

Fig. 10 - PH [100], completely recrystallized showing shingle-like structure at the grain boundary, highly etched.

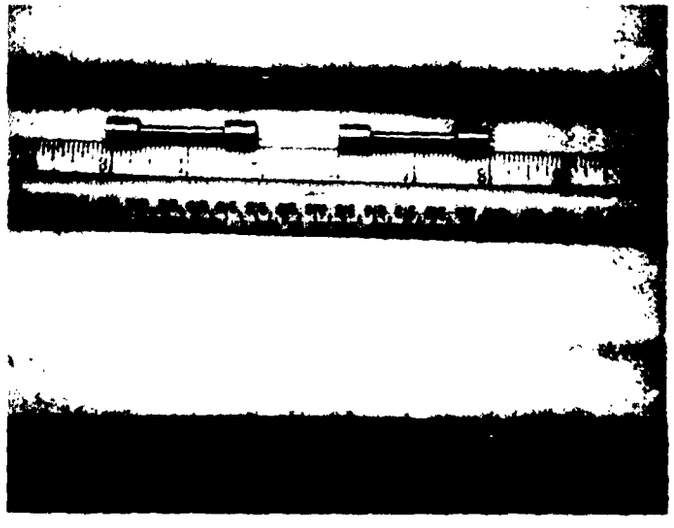


Fig. 11 - A section of swaged rod and two tensile specimens prepared from a similar rod.

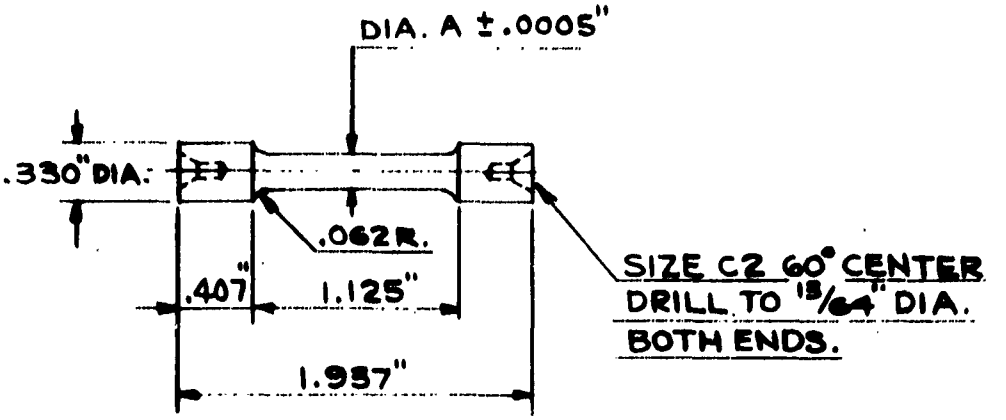


FIGURE 12a - DIMENSIONS OF TENSILE SPECIMEN OBTAINED FROM SWAGED ROD.

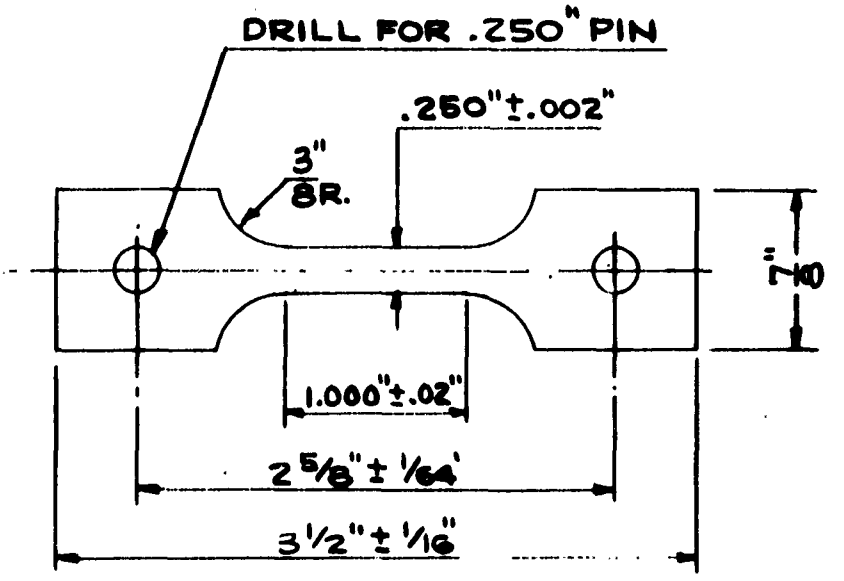


FIGURE 12b DIMENSIONS OF TENSILE SPECIMEN OBTAINED FROM ROLLED SHEET.

Fig. 13 - Photo of cleaved tungsten, the side to the upper right has been ground. Note the smooth planar effect.

10X

Fig. 14 - Photo of fracture along boundaries. Note the curved and uneven appearance.

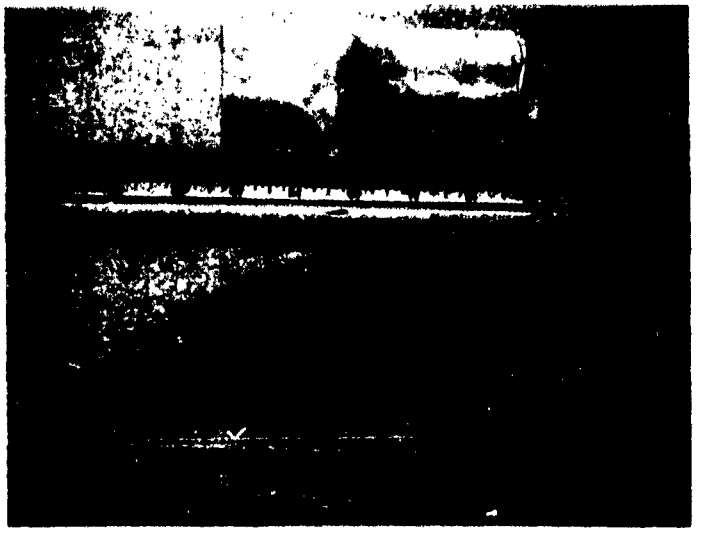
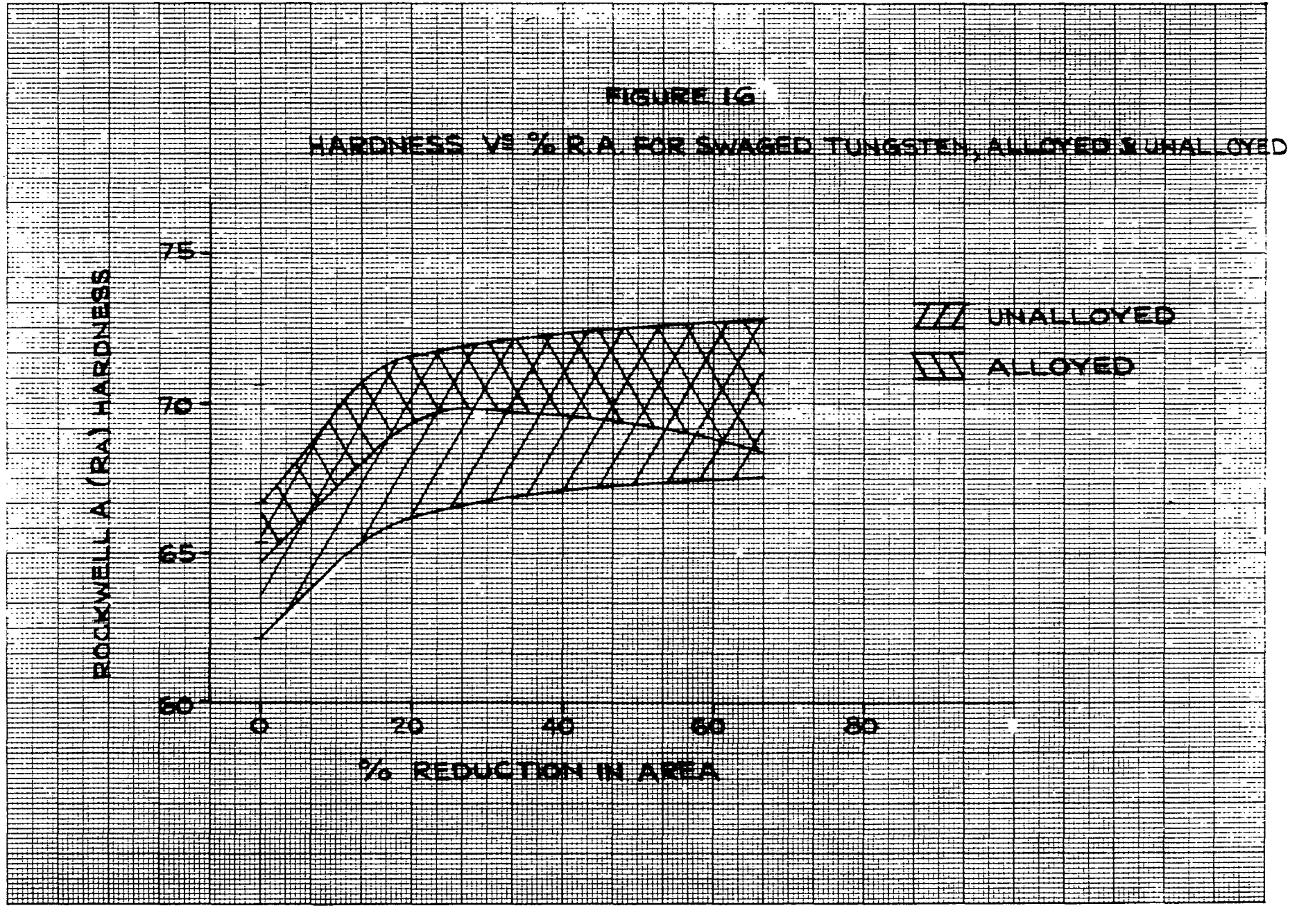
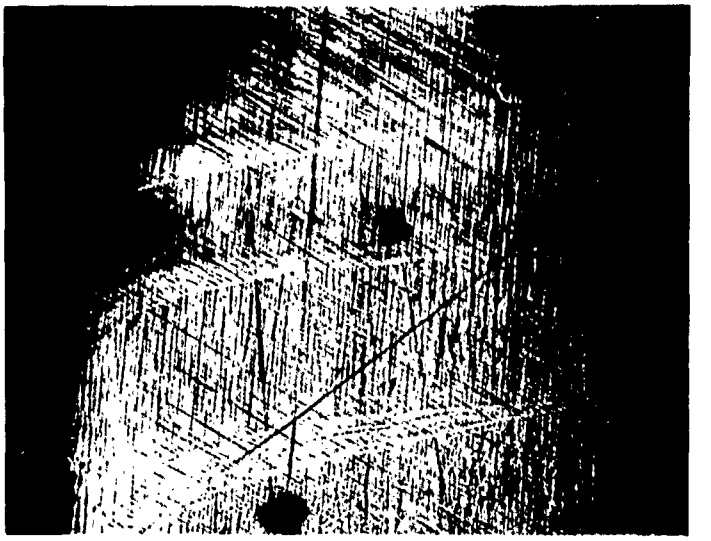


Fig. 15 - Some sections of worked material. Two pieces of 0.040" sheet and one swaged rod.

FIGURE 16

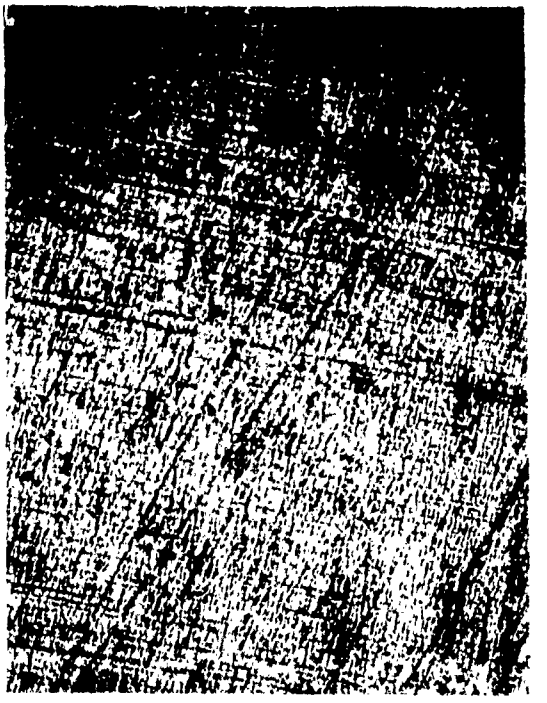
HARDNESS Vs % R.A. FOR SWAGED TUNGSTEN, ALLOYED & UNALLOYED





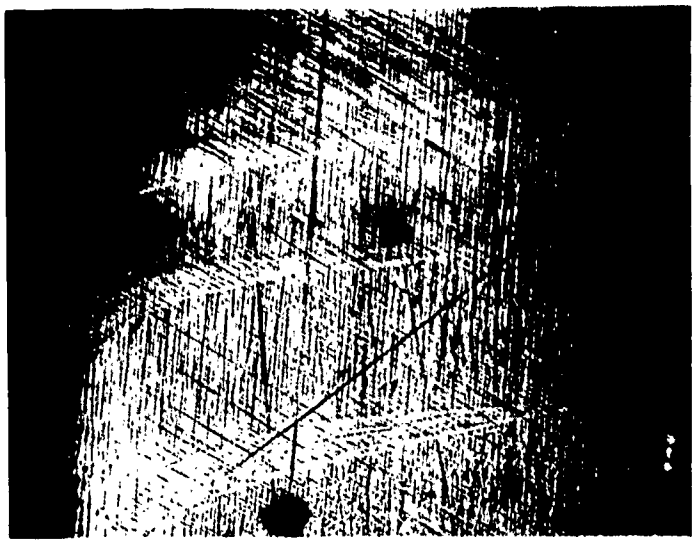
50X

Fig. 17a - PAB [111], a section of an unalloyed tungsten crystal swaged to 20% R.A. Note the presence of deformation bands.



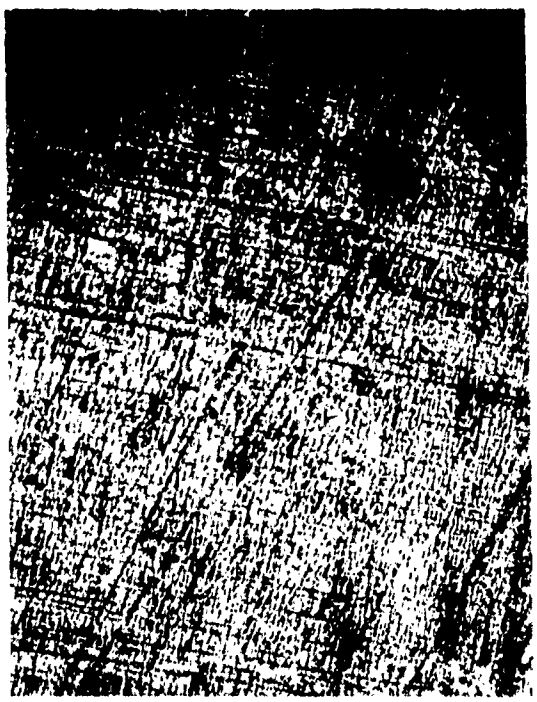
50X

Fig. 17b - PAB, same as Fig. 17a, vacuum annealed at 1300°C for 1/2 hour.



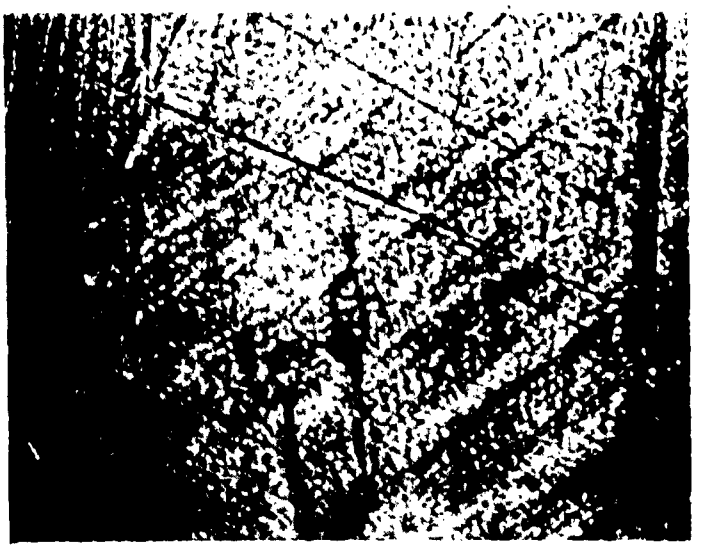
50X

Fig. 17a - PAB [111], a section of an unalloyed tungsten crystal swaged to 20% R.A. Note the presence of deformation bands.



50X

Fig. 17b - PAB, same as Fig. 17a, vacuum annealed at 1300°C for 1/2 hour.



50X

Fig. 17c - PAB, same as Fig. 17a, vacuum annealed at 1500°C for 1/2 hour.

500X

Fig. 17d - PAB, same as Fig. 17a, vacuum annealed at 1500°C for 1/2 hour. Note the start of recrystallization.

50X

Fig. 17e - PAB, same as Fig. 17a, vacuum annealed at 1650°C for 1/2 hour.

50X

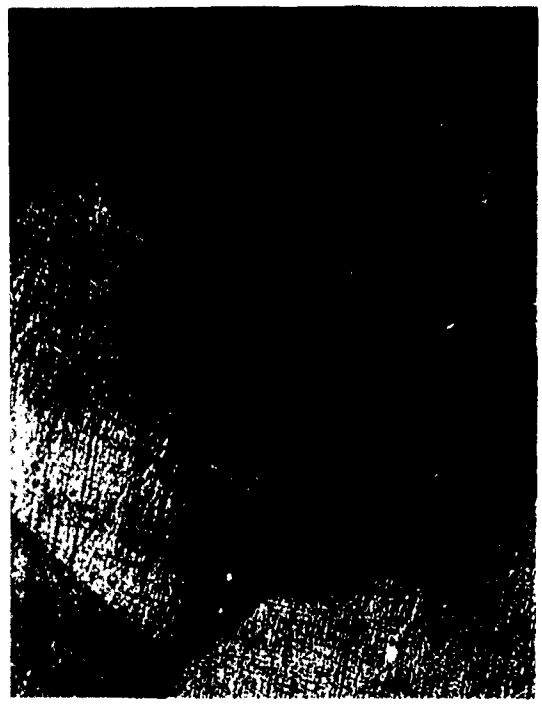
Fig. 17f - PAB, same as Fig. 17a, vacuum annealed at 1725°C for 1/2 hour.

3X

Fig. 17g - PAB, same as Fig. 17a, vacuum annealed at 1725°C for 1/2 hour.

3X

Fig. 17h - PAB, same as Fig. 17a, vacuum annealed at 1800°C for 1/2 hour.

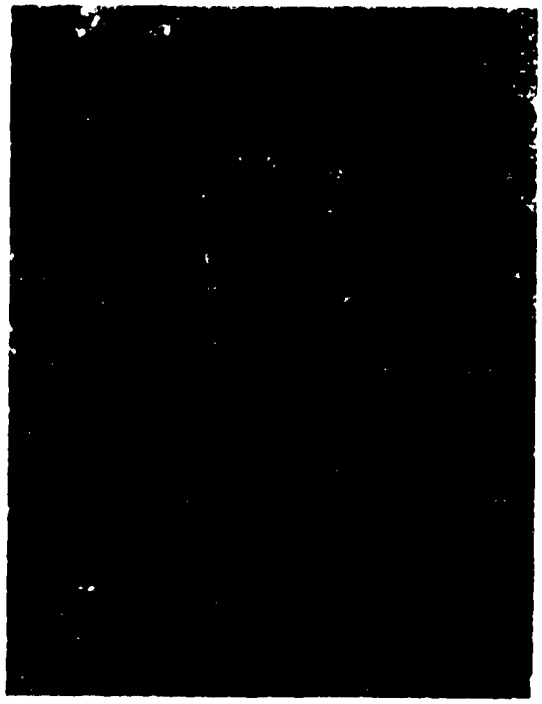


50X

Fig. 17i - PAB, same as Fig. 17a, vacuum annealed at 2000°C for 1/2 hour. Note recrystallization is complete.

3X

Fig. 17j - PAB, same as Fig. 17a, vacuum annealed at 2000°C for 1/2 hour.



150X

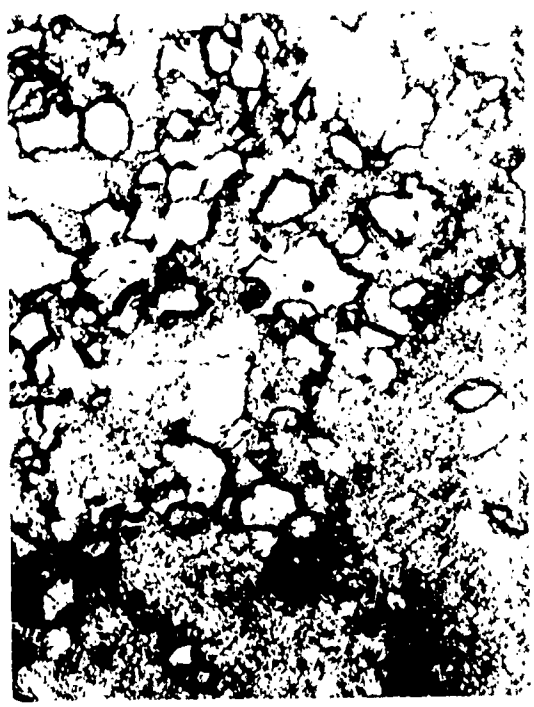
Fig. 18a - RU-1 [111], a section of an 1% Nb alloy forged from .505 inch to .230 inch and rolled to .040 inch. Refer to Fig. 21 which is at a lower magnification and one can see the presence of deformation bands.

150X

Fig. 18b - RU-1, same as Fig. 18a, vacuum annealed 1350°C for 1/2 hour.

500X

Fig. 18c - RU-1, same as Fig. 18a, vacuum annealed at 1350° for 1/2 hour.



150X

Fig. 18d - RU-1, same as Fig. 18a, vacuum annealed 1500°C for 1/2 hour.

150X

Fig. 18e - RU-1, same as 18a, vacuum annealed 1620°C for 1/2 hour.

150X

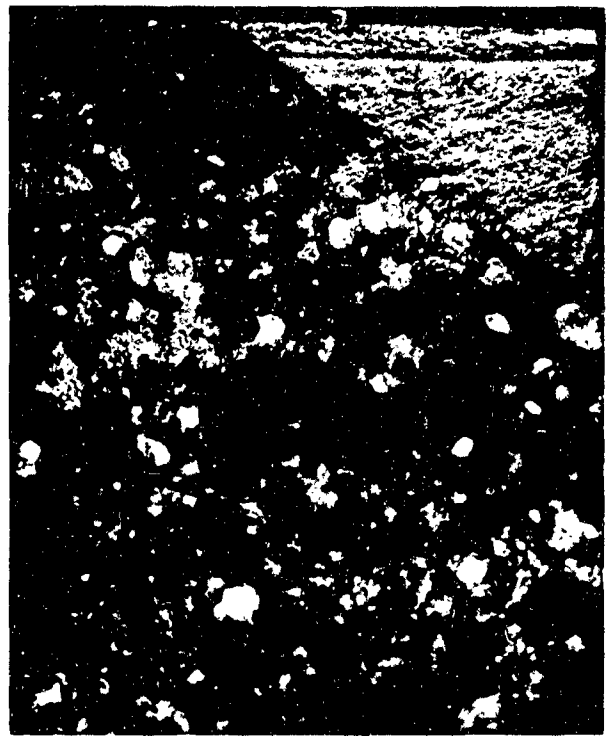
Fig. 18f - RU-1, same as Fig. 18a, vacuum annealed 1775°C for 1/2 hour. Very heavy etch.

150X

Fig. 18g - RU-1, same as Fig. 18a, vacuum annealed 1850°C for 1/2 hour.

150X

Fig. 18h - RU-1, same as Fig. 18a, vacuum annealed 1975°C for 1/2 hour.



10X

Fig. 18i - RU-1, same as Fig. 18a, vacuum annealed 1975°C for 1/2 hour. Note that some coarsening has taken place. The unetched portion is where the electrical contact was made.

150X

Fig. 18j - RU-1, same as Fig. 18a, vacuum annealed 2100°C for 1/2 hour. Note that recrystallization is complete.

FIGURE 19a

ROOM TEMPERATURE HARDNESS VS TEMPERATURE, ½ HOUR ISOCHRONAL ANNEALS, FOR WORKED UNALLOYED TUNGSTEN (TYPICAL CRYSTALS)

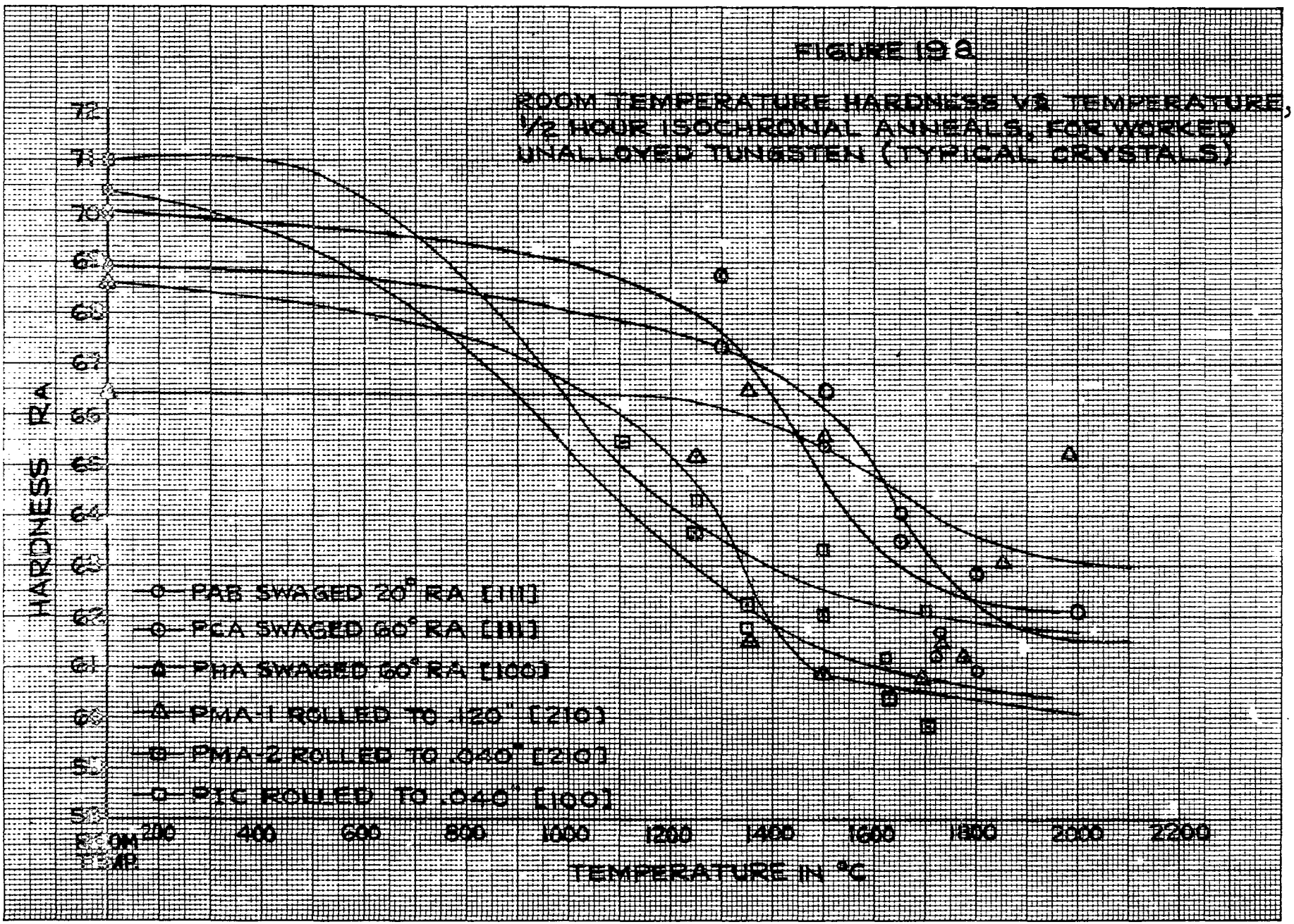


FIGURE 19b

ROOM TEMPERATURE HARDNESS VS TEMPERATURE, 1/2 HOUR ISOCHRONAL ANNEALS, FOR WORKED TUNGSTEN ALLOYS ALL [111]

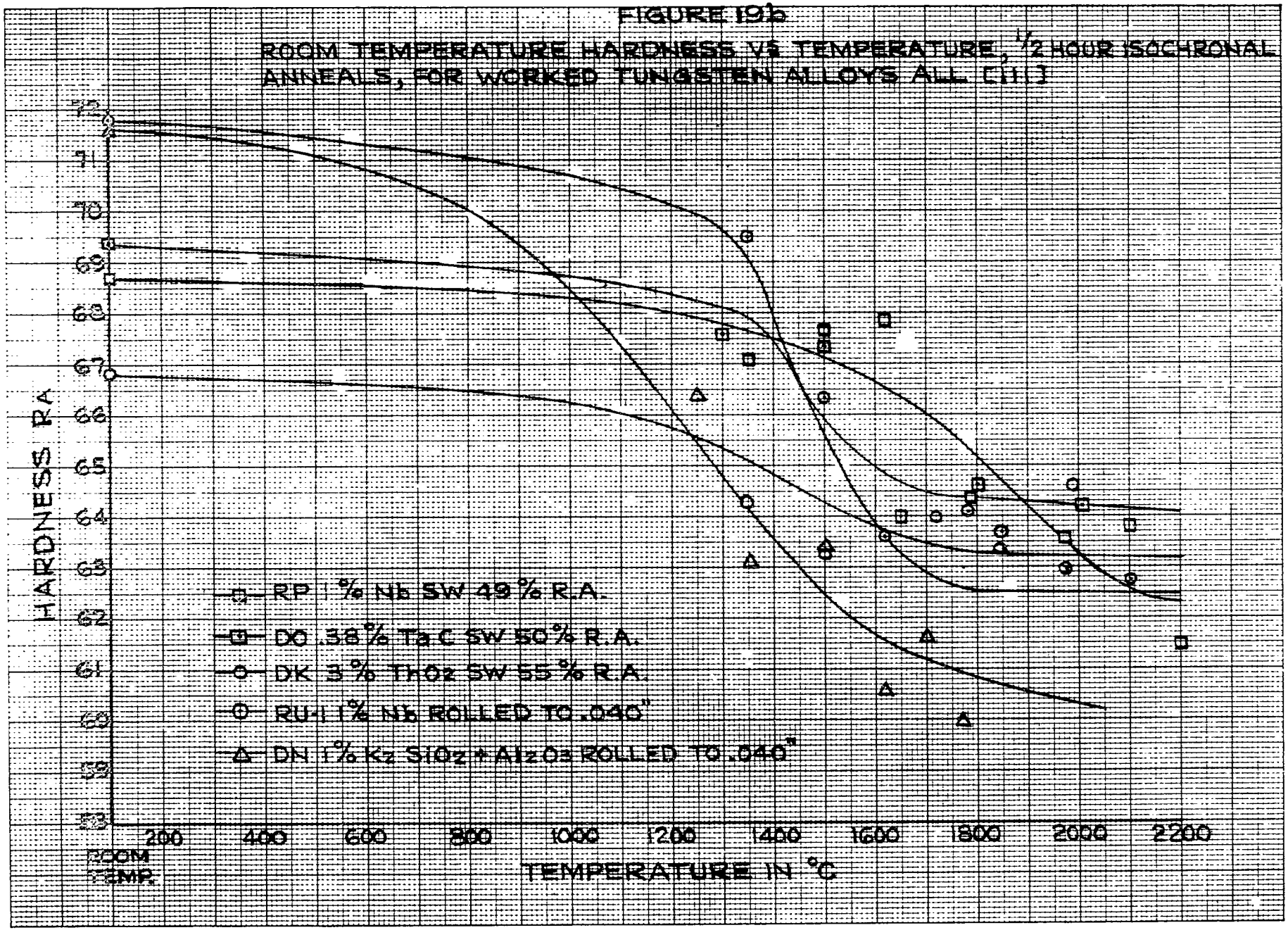


FIGURE 203

ESTIMATED % OF MICROSTRUCTURE WHICH HAD RECRYSTALLIZED AS DETERMINED METALLOGRAPHICALLY VS. TEMPERATURE FOR WORKED UNALLOYED TUNGSTEN (TYPICAL CRYSTALS)

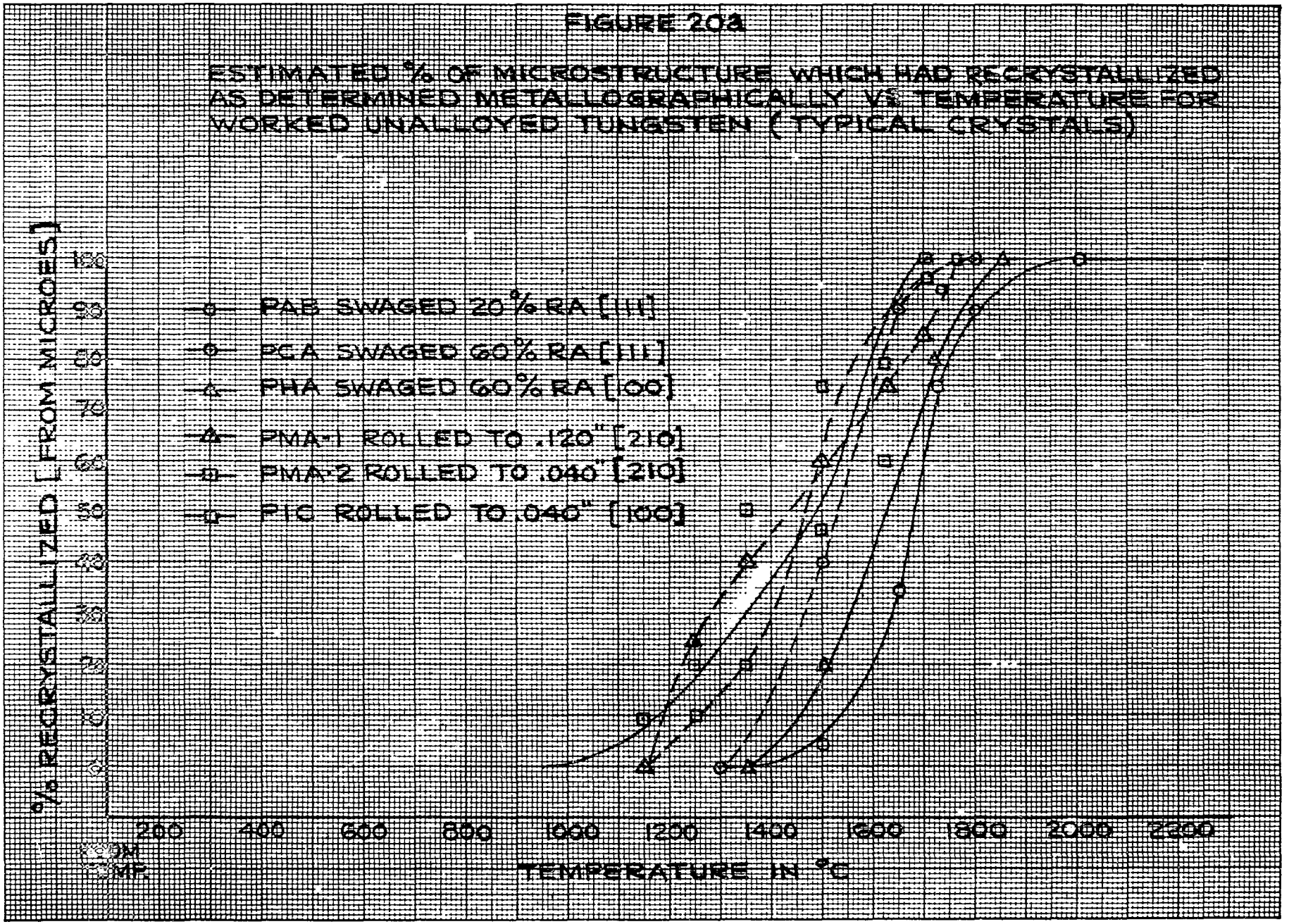
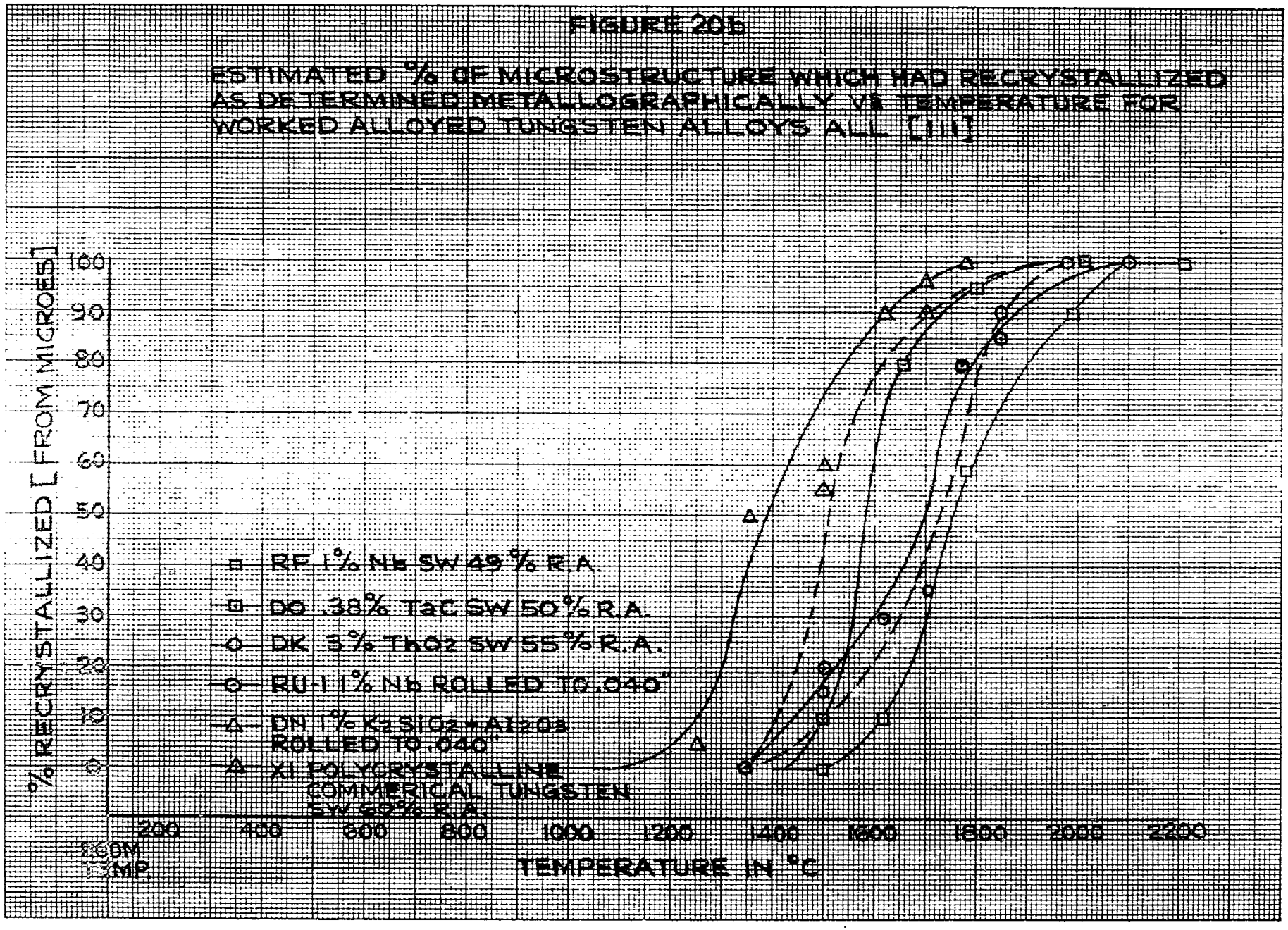


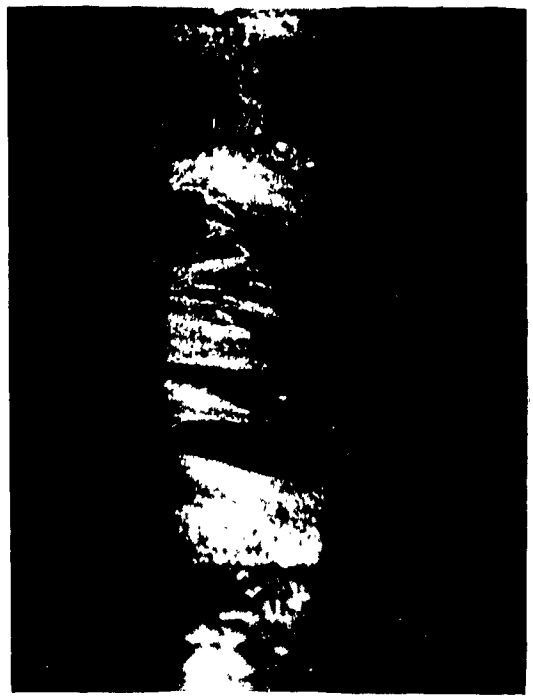
FIGURE 20b

ESTIMATED % OF MICROSTRUCTURE WHICH HAD RECRYSTALLIZED AS DETERMINED METALLOGRAPHICALLY VS TEMPERATURE FOR WORKED ALLOYED TUNGSTEN ALLOYS ALL [111]



3X

Fig. 21 - RU-1 [111], 1% Nb alloy forged from .505 inch to .230 inch and rolled to .040 inch. Note the presence of deformation bands.



6X

Fig. 22 - PGC [111], unalloyed tungsten crystal. This is the side view after being forged from .580 inch to .256 inch. Note the presence of deformation bands.

6X

Fig. 23 - PCB [111], unalloyed tungsten crystal swaged to 60% R.A.

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